TEXTILE RESEARCH JOURNAL

VOL. XV, NO. 3 March, 1945

Applications of Nitrogen Dioxide Treatment to the Microscopy of Fiber Cell Wall Structure

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DURING the course of studies in this laboratory on the oxidation of cellulose, air-dried cotton fibers were treated with gaseous nitrogen dioxide using the cyclic method described in 1942 by Yackel and Kenyon [43].[1] According to the original work, the fibrous material obtained by this method contains no nitrogen but differs from the original cotton in that part or all of the primary hydroxyl groups of the cellulose molecule are oxidized to carboxyl. A notable property of cellulose which has been so treated is its rapid and complete solubility in dilute alkaline solutions.

Microscopical examination of the fibers was made to determine the extent of fiber damage. They were found to be much swollen (Figures 1 and 2) and severely weakened but with spiral patterns (Figure 3) on the surface of the fiber more prominent and distinct than had been previously observed in microscopical studies of cell wall structure. Accordingly, preliminary swelling experiments were initiated to

FIG. 1. *Longitudinal view of Upland cotton fibers. Top—Untreated. Bottom—NO_2-treated 20 hours. Magnification 125 ×.*

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[1] Numbers in brackets indicate references to literature cited at the end of this paper.

observe details of fiber structure brought out by the nitrogen dioxide treatment. The accompanying photomicrographs show fibers which have received somewhat drastic treatment and do not represent the natural condition of the cell wall. They do, however, appear to offer a new method of approach to investigations of the fine structure of fibers. Further application of the NO_2-oxidation technique to the microscopical study of fibers should prove valuable in the evaluation of commercial treatments of fibers.

Previous Work

The literature on microscopical studies of cell wall structure is voluminous, and no attempt is made here to review it. Suffice it to say that since the work of Nageli [29] (1855) micrographs and photomicrographs have been published of various native fibers in an attempt to explain the structural peculiarities of the natural fiber, and in some degree to correlate the microscopical pattern with the chemical evidence and x-ray data. A fairly complete bibliography is contained in a review article by R. F. Nickerson [30], and thorough discussions are presented by C. W. Hock in the "Symposium on the Structure of Protoplasm," edited by Seifriz [27]; and by Hock, Mark, and Sears in Chapter III C of "Cellulose and Cellulose Derivatives," edited by Emil Ott [33].

By far the most extensive work has been done on cotton [3, 4, 11, 12, 13, 19, 32, 35] but enough has been published on flax [2, 31], jute [34], ramie [18], valonia [5, 42], and wood fibers [22, 23, 36] to indicate a certain fundamental similarity in the structure of native cell walls, from whatever source.

The ultimate fiber unit is a single cell which may be roughly divided into three regions: the thin primary wall which delimits the length of the fiber; a secondary wall, which, in cotton, at least, has been shown to grow by apposition in diurnal layers deposited centripetally from the outside cell wall toward the center of the fiber; and the lumen or central canal which in the living cell is filled with protoplasm and in the dried cell is hollow except for the desiccated protoplasmic residues.

It is generally conceded that in all vegetable fibers (wood, seed hairs, leaf, and bast) the primary wall exists as a distinct entity, and that it has certain physical and perhaps chemical characteristics that differ markedly from those of the secondary wall. In cotton the primary wall is believed to be made up of a network of branching cellulose fibrils which spiral about the fiber at an angle of from 10 to 90 degrees with the fiber axis. Anderson and Kerr [3] and Hock, Ramsay, and Harris [26] have photographed these in young cotton fibers which were stained with Congo red or chlorozinc iodide and examined between crossed Nicols. There appear to be two systems of spiral threads forming right- and left-hand spirals in the primary wall at an angle of 70 degrees with the axis of the fiber. Anderson and Kerr indicate that there may also be a third system of threads in the primary wall which make a 90-degree spiral about the fiber. None of these structures is visible except when the fiber is stained with a dichroic reagent and viewed between crossed Nicols; nor are they seen in any but very young fibers. Apparently, after secondary wall growth has begun at the age of 16 days the primary wall structure is so masked by the secondary wall orientation that details of the primary wall are difficult to observe.

In cotton the cellulose network of the primary wall is embedded in a matrix of waxes and pectic materials which, upon proper treatment, can be dissolved to leave the cellulose skeleton. The outermost layer of the waxy coating is referred to as the cuticle of the fiber.

The secondary wall is composed of many sleeve-like layers which in cross section appear as concentric cylinders. In cotton these have been correlated with age in days of growth and have been likened to the "growth rings" of forest trees. The laminated structure of the secondary wall has been most clearly shown by Kerr [28] in excessively swollen cross sections of fibers which appear to have distinct concentric layers. Lamination has also been shown [26] in longitudinal views of swollen fibers, as in Figure 4. Each layer is considered [3, 28] to consist of two portions, one compact and the other porous, together constituting a daily growth ring in the development of the fiber. Each lamella is thought to be further divided into tiny thread-like components called fibrils, which are aligned side by side, anastomosing from time to time as they follow a spiral path about the longitudinal axis of the fiber; but whether these spirals occur in both the porous and the compact layers is not clear. Between the thin, film-like primary wall and the many-layered secondary wall lies a region of the cotton cell wall which seems to differ from both. This layer has been called the "first layer of the secondary wall" because it is the first layer of cellulose laid down in which crystalline orientation of the cellulose can be detected in x-ray studies. It might also be called the "inner layer of

the primary wall" because it separates so cleanly from the rest of the secondary wall upon stretching or swelling of the fiber, and because in the mature fiber it can be separated from the primary wall only with the greatest difficulty.

Anderson and Kerr [3] point out that this first deposited layer of secondary cellulose is made up of coarse threads whose spiral angle is approximately 20–30 degrees with the fiber axis, in contrast to the 70-degree angle of the right- and left-hand spirals of the primary wall. They also state that the direction of this spiral may reverse at random throughout the length of the fiber. Subsequent layers of the secondary wall are composed of much finer threads, which may spiral in an opposite direction, or often in the same direction as the coarse threads of the first layer, and may have the same angle or a much steeper angle than that of the first layer of the secondary wall. Hock, Ramsay, and Harris [26] describe the secondary wall as composed of innumerable fine fibrils enclosed by a "winding" which appears to be coarser than the fibrils themselves. They find that invariably the orientation of fibrils immediately beneath the "outer winding" is in the opposite direction to that of the "winding" and that where reversal of fibrils in the "winding" occurs a corresponding reversal in all the layers of the secondary wall occurs, but in the opposite direction. These reversal points are held to be responsible for the optical differences noted in polarized light where bands of extinction occurring along the fiber length are places at which reversals occur. An attempt is also made to correlate the direction of spiral windings with the convolutions of the cotton fiber. Obviously the "winding layer" of Hock, Ramsay, and Harris and the "first layer of secondary thickening" of Anderson and Kerr are identical. It may be considered almost as a transition layer between the primary wall of the young cell which shows no crystalline orientation by x-ray analysis, and which is impregnated with waxes, pectin, and other noncellulosic materials, and the highly oriented, relatively pure cellulose of the many-layered secondary wall. Together, the primary wall and the "first secondary layer" (or "winding layer") form an outer protective sheath about the remainder of the fiber, much like the cortex of a woody stem. In fact, we might think of the primary wall as the "outer sheath" and the "winding layer" as the "inner sheath" of this skin-like membrane.

In wood fibers Hock and Seifriz [25], Ritter [36, 37, 38], Bixler [16], I. W. Bailey and Kerr [6, 8, 9], and others have shown spiral strands in the primary wall, and the sleeve-like layers of which the secondary wall is composed. I. W. Bailey and Vestal [10] have demonstrated, by the deposition of iodine crystals in longitudinal sections of conifer tracheids, the angular arrangement of cellulose fibrils in certain inner wall layers and the longitudinal arrangement of fibrils in other layers of the same cell walls. In flax, both Nodder [31] and Anderson [2] have shown spiral fibrils in the outer cell wall like those shown by Preston [34] in jute; and similar helices are demonstrated in ramie by swelling and pressure techniques in the work of Eisenhut and Kuhn [18]. Astbury and his co-workers [5] in studying the wall of the single-celled alga, valonia, found it to be composed of several layers of spirally arranged cellulose strands in a pattern somewhat similar to Anderson and Kerr's diagram of the cotton fiber. The work of Farr [20] and her co-workers indicates that the fibrils of the cell wall of cotton are composed of strings of cellulose particles held together by cementing substances in the cell wall. However, the particle size as measured in this work has been severely criticized [3] in the light of other cell wall dimensions.

The work of Farr and Clark [21] and others suggests the use of polarization colors as an index of the degree of regularity of orientation of structural units, as well as the study of cell wall orientation by means of x-ray diffraction patterns. However, it was pointed out in the paper by I. W. Bailey and Vestal [10] that phenomena observed under the polarization microscope are significant only when material can be oriented so that the polarized light passes through single layers, but that accurate interpretations are difficult when the light passes through several superimposed layers of varying thickness and of different fibrillar orientation. They further conclude that similar obstacles must be overcome in the interpretation of x-ray diagrams where the rays pass through a complex of layers of markedly varying thickness and of very different structural orientations.

Sisson [40, 41, 42], using x-ray diffraction, has measured the crystalline orientation of various vegetable fibers, and has indicated that there is a correlation between the angle of fibril orientation as observed microscopically and the crystalline orientation measured in x-ray diffraction patterns. Sisson evidently recognized the difficulty in such correlation: "The usual x-ray diagram represents the average orientation throughout a millimeter section of several thousand fibers, while microscopic results are specific

for localized sections of single fibers" [41]. Unfortunately, later discussions of this work [33] have implied a more specific correlation between observed microscopic organization and crystalline orientation by x-ray measurement than the evidence presented seems to warrant. I. W. Bailey and Berkley [7], in work on the relatively simple structure of conifer tracheids, report that fibrillar orientation can best be studied microscopically in individual layers of the cell wall after deposition of crystals in the cellulose matrix. They found that fibrillar orientation of cellulose in the secondary walls of tracheids fluctuate markedly from layer to layer, and even in different lamellae of the same layer. They conclude that the over-all average or dominant arrangement of the crystalline cellulose may best be obtained from x-ray diffraction patterns but the use of the polarizing microscope and of x-ray diffraction patterns alone may lead to serious misconceptions about the orientation of cellulose in cell walls, unless the numerous morphological, histological, and chemical variables are accurately visualized and accounted for.

Materials and Methods

Much of the previous work in the field of cell wall structure has been done on living or fresh fibers, following the ontogeny and growth of the cell from its inception to maturity. Such studies on developmental material have contributed much to our knowledge of growth processes. However, for application of microscopical techniques to laboratory control procedures in the processing of cotton yarns and fabrics, it is necessary that we study the appearance and construction of the mature fiber as it occurs in commerce. Thus only may the microscopist be able to assist the chemist in evaluating the effect of experimental treatments and of commercial practices in the processing of fibers for manufacture.

Commercial samples of Chinese cotton and an American Upland cotton of middling grade were used for a part of these studies. Cotton fibers of Mexican Big Boll cotton, 24 days after bloom, and 42 days after bloom, from seeds preserved in alcohol,* were also used. Tentative studies were made on bleached ramie fiber, unbleached flax fiber, unbleached jute, and unbleached milkweed bast fiber, in addition to cotton.

Air-dried fibers which had been subjected to NO_2 gas for varying lengths of time were washed

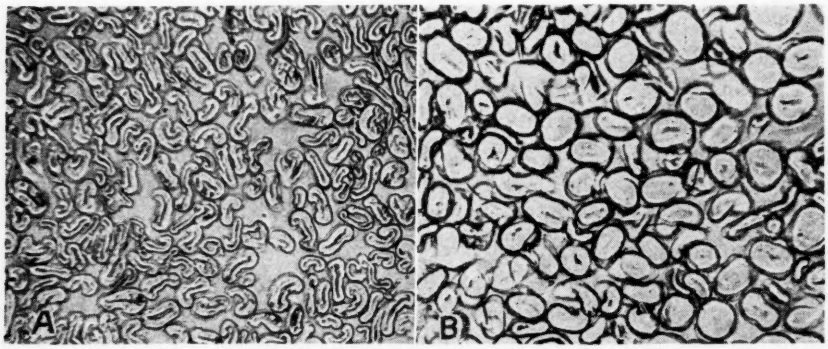

FIG. 2. *Cross section of Upland cotton fibers. A—Untreated. B—NO_2-treated for 20 hours. Magnification 250 ×.*

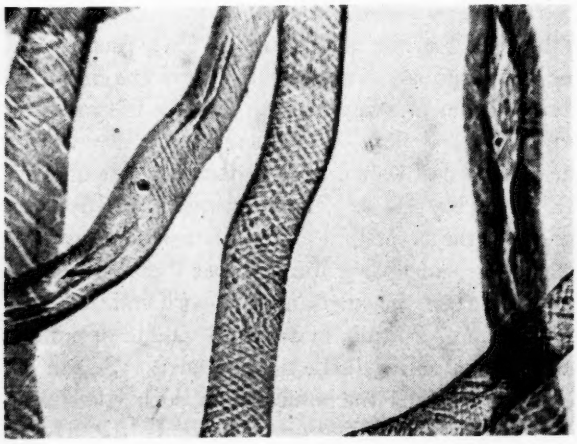

FIG. 3. *Chinese cotton, NO_2-treated 16 hours, swelled with cuprammonium hydroxide. Magnification 190 ×.*

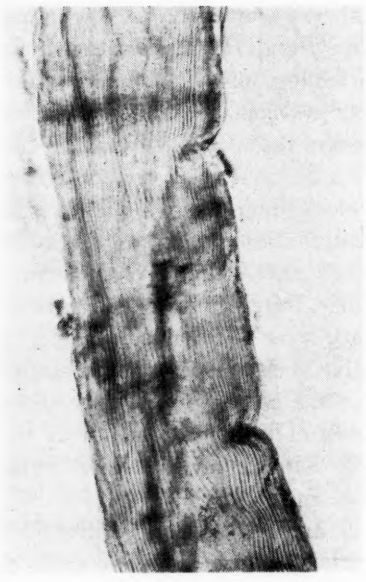

FIG. 4. *Cotton fiber from commercial cord, swelled in 1[illegible] percent NaOH and cuprammonium hydroxide. Note parallel layers of secondary wall. Magnification 250 ×.*

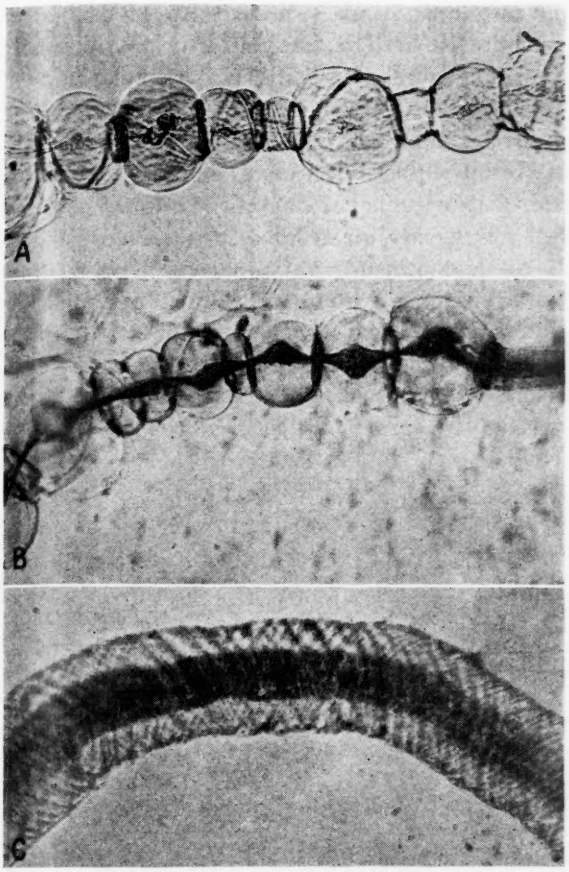

FIG. 5. *A and B—Chinese cotton, untreated, swelled with cuprammonium hydroxide. Note balloons, broken sheath, and twisted lumen. Magnification 250 ×. C—Chinese cotton, NO_2-treated 16 hours, swelled with cuprammonium hydroxide. Note fine fibrils of sheath. Magnification 500 ×.*

dried, and examined microscopically while mounted in water. To bring out the finer structural details of the oxidized fiber, swelling agents were introduced under the cover slip. The usual reagents were employed, including sodium hydroxide, trimethylbenzylammonium hydroxide, ammonia, and cuprammonium hydroxide. After treatment with nitrogen dioxide, fibers swell appreciably in any dilute alkaline solution. The most satisfactory reagent was found to be cuprammonium hydroxide of the composition specified by the Committee on Viscosity of Cellulose of the American Chemical Society [1] (30 grams of copper and 165 grams of ammonia per liter) diluted to about one-third or one-sixth strength. The fibers were stained with ruthenium red, methylene blue,

* Obtained from Dr. E. E. Berkley, Bureau Plant Industry, Soils, and Agricultural Engineering.

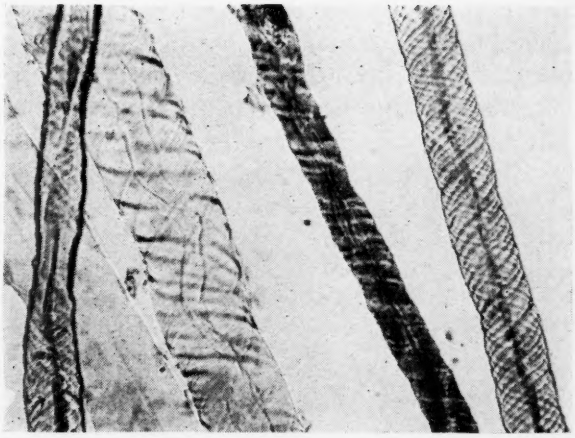

FIG. 6. *Forty-two-day-old fiber of Mexican Big Boll cotton; NO_2-treated 1 hour; swelled in NaOH (1 percent). Note 5 different stages in swelling. Magnification 200 ×.*

Congo red or safranine, but for photographic purposes the most satisfactory stain was found to be a 50–50 mixture of 1 percent aqueous solutions of methylene blue and safranine.

Experimental Results

When raw cotton fibers are swelled with cuprammonium hydroxide, "balloons" form wherever the primary wall breaks to allow swelling of the cellulose in the layers of the secondary wall beneath it (Figures 5A and 5B). By contrast, in fibers treated with NO_2, rupture of the fiber sheath is more uniform and balloons do not develop; instead, the spiral splitting of the sheath occurs more or less uniformly throughout the length of the fiber (Figure 5C). The primary wall is distinctly different from the secondary wall in its reaction toward stains and swelling agents. The lumen behaves like the primary wall in both respects (Figure 5B). Ruthenium red imparts a fuchsia color to both the lumen and the primary wall; methylene blue stains them a deep blue-purple, and safranine stains them brown-to-gold in alkaline solution. The structural pattern made by rupture of the sheath varies considerably under the same conditions of swelling. Figures 6 and 7 show types of fibril patterns produced by swelling after treatment with NO_2. Patterns such as those in Figure 8 raise the question of whether reversal of fibril direction in the primary wall is a reality or an artifact due to inequalities in swelling. Other workers [3] have indicated that reversal of fibril direction never occurs in the primary wall but only in the secondary wall

layers. If the NO_2 treatment has been brief—a few minutes to an hour—the fiber sheath is seen to split into many fine fibrils (Figures 6 and 9). If the NO_2 treatment has been prolonged the sheath may split into broad bands (Figure 11A), or flake off, as in Figure 10A. The unusual views of cotton fibers shown in Figures 11A and 11B were obtained by the excessively long treatment of 74 and 72 hours respectively, after which the major part of the fiber was readily dissolved in the swelling agent while an outer portion remained more or less intact as a solid shell. Evidently the cellulose of the secondary wall was so deteriorated by the prolonged NO_2 treatment that no swelling pressure was exerted by it to rupture the fiber sheath, and it would appear that the sheath might have undergone some "curing" effect, which rendered it unusually resistant. In a moderately

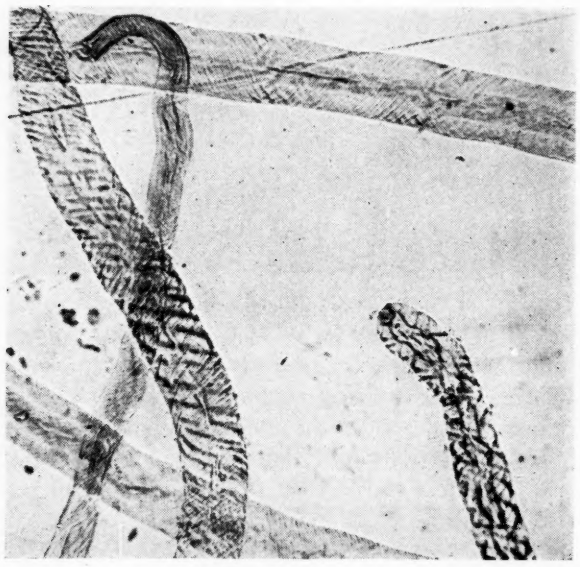

FIG. 7. *Chinese cotton NO_2-treated 16 hours; swelled with cuprammonium. Magnification 250 ×.*

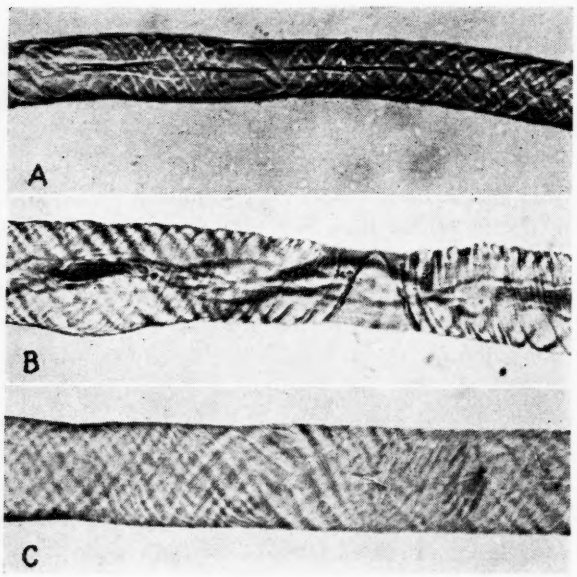

FIG. 8. *Structure of fiber sheath. A—Chinese cotton, NO_2-treated 16 hours; swelled with N/50 NaOH. B—42-day-old Mexican Big Boll cotton; NO_2-treated 1 hour; swelled with 1 percent NaOH. C—Chinese cotton, NO_2-treated 16 hours; swelled with cuprammonium hydroxide. Note "reversals" of fibrils. Magnification 250 ×.*

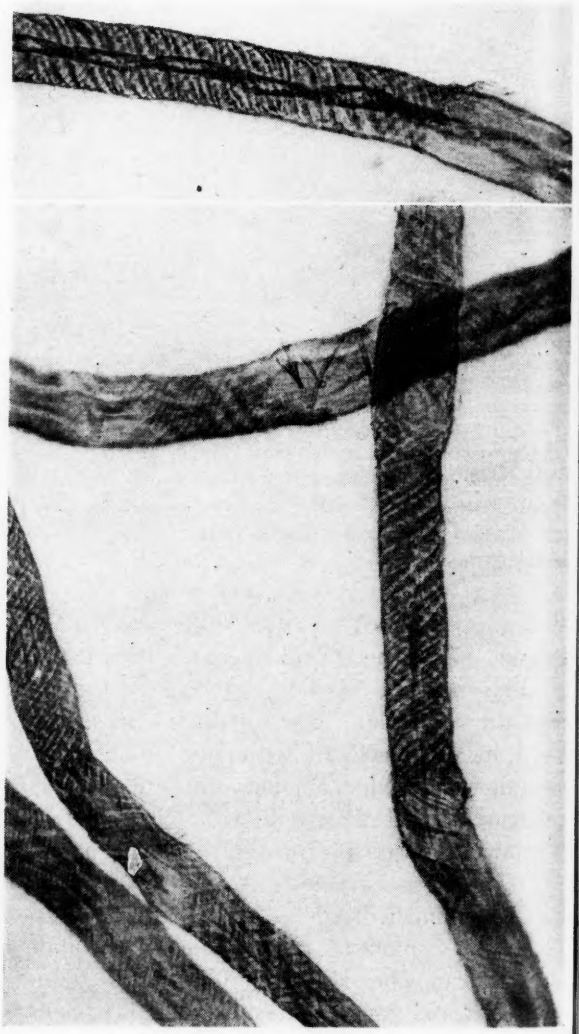

FIG. 9. *Forty-two-day-old Mexican Big Boll cotton fiber; NO_2-treated 1 hour; swelled with 0.1 percent NaOH. Note peeling off of fiber sheath. Magnification 250 ×.*

oxidized fiber drastic swelling occurs when a strong swelling agent is applied (Figure 12).

Mature cotton treated for an hour with NO_2 and swelled in lyotropic solutions undergoes progressive swelling in such a series as is shown in Figures 13, 14, and 15. The unswelled portion of the fiber protected by the primary wall stains a dark purple with methylene blue. The broken sheath can be seen separating into a spiral which wraps around the fiber (Figure 14A). The spirals are dark blue-purple and the pale blue-to-colorless parallel layers of the secondary wall can be plainly seen inside the spirals. No amount of focusing up or down through the thickness of this specimen, even at 1,200 magnifications, revealed any semblance of spiral lines in any of the various layers of the secondary wall. It is possible that they have been fused by the swelling treatment.

Figure 15 shows the frayed broken ends of such fibers as those shown in Figures 13 and 14. This disintegration developed as the result of prolonged swellings, but without pressure or other manipulation of the fibers. The parallel layers of the cellulose

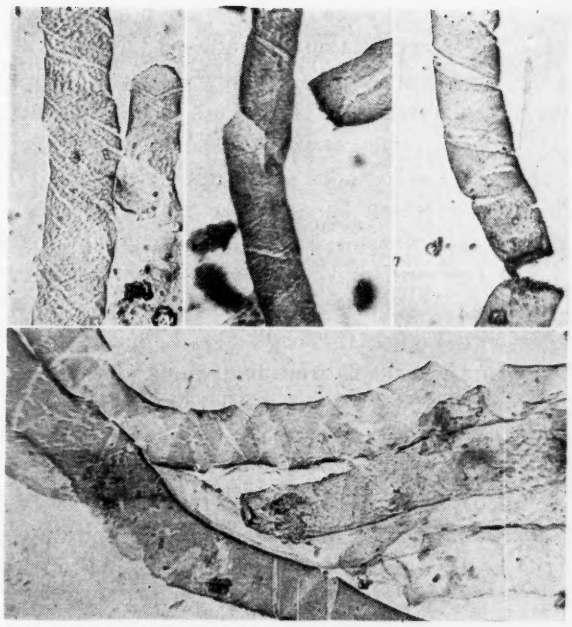

FIG. 11. *A—Chinese cotton, NO_2-treated 74 hours; swelled in cuprammonium hydroxide. Note persistence of fiber sheath after solution of secondary wall. 190 ×.*

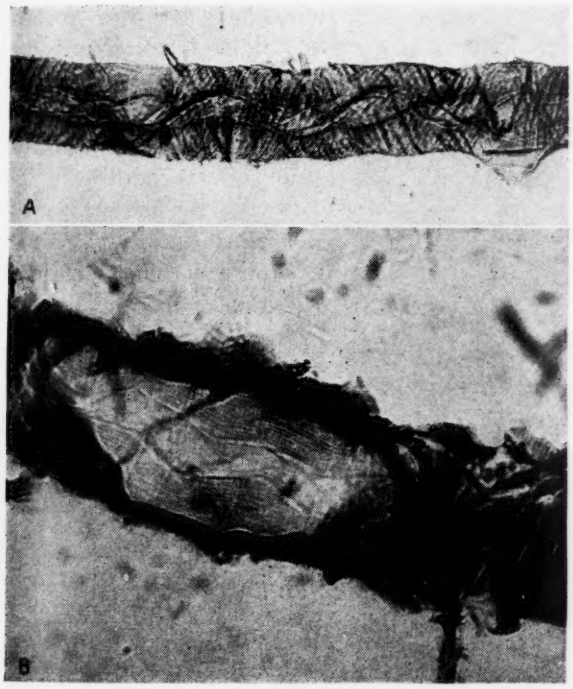

FIG. 10. *Forty-two-day-old Mexican Big Boll cotton fiber. A—NO_2-treated 6 hours; swelled with NH_4OH. Note peeling of fiber sheath. B—NO_2-treated 1 hour; swelled with 1 percent NaOH. Note parallel layers of secondary wall beneath peeling fiber sheath. Magnification 190 ×.*

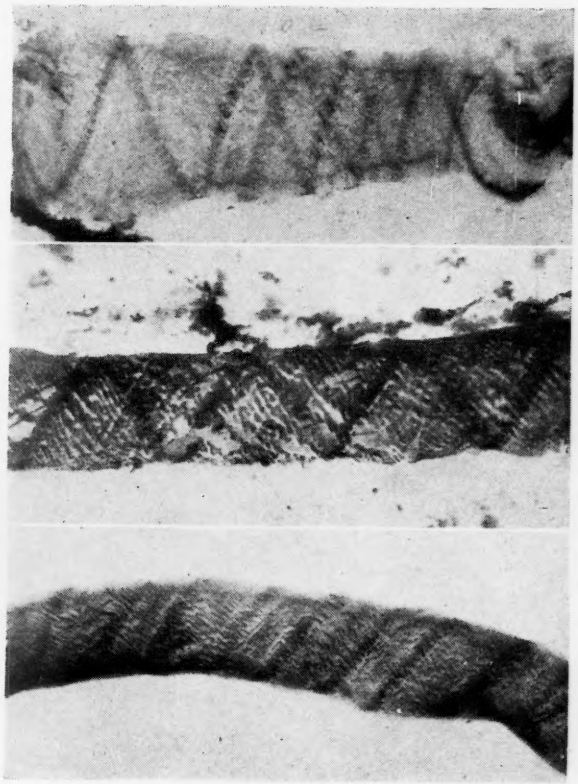

FIG. 11. *B—Chinese cotton, NO_2-treated 72 hours; swelled in dilute ammonium hydroxide. Note banded structure of fiber sheath. Magnification 190 ×.*

walls can be readily seen breaking up into finer units. While the magnification in Figure 15 is only 200, the enlargement due to swelling is such that the over-all enlargement of the fibers is approximately 700. At this point comparison should be made with the electron micrographs of Barnes and Burton [14] which show cotton fibers beaten into fragments by mechanical action in water, and magnified 6,000 times. The structural relationships seem evident by comparison of Figure 6 from the Barnes and Burton paper with Figure 15 (page 74). Similar electron micrographs of fibrils from both rag and wood pulp are shown by Sears and Kregel [39] in their publication on the use of the electron microscope in the paper industry.

While none of the photomicrographs obtained from NO_2-treated cotton fibers confirm the spirality of fibrils in the secondary wall lamellae as indicated by Anderson and Kerr's cell wall diagram [3], Berkley's schematic drawing [15], and Ritter's discussion of wood cell walls [36], it has been possible to photograph in ramie (Figure 16) and in milkweed bast fibers (Figure 17) spiral fibrillar structures in the secondary wall which correspond to those described for cotton. These compare favorably with the photomicrographs by Bailey and Vestal [10] showing the orientation of secondary wall fibrils in wood tracheids as demonstrated by the deposition of iodine crystals in their interstices. It is possible that the spirals in the secondary wall lamellae of cotton are so much finer than those of the inner sheath that they are affected by the drastic treatment the fiber has undergone in NO_2 oxidation.

In Figure 16B the angle which the fibrils of the secondary wall of ramie make with the longitudinal axis of the fiber varies from approximately 20 to 34 degrees, whereas that of the spirals in the primary wall of Figure 18B varies from approximately 28 to 45 degrees. Similarly, the angle for fibrils of the primary wall of flax if measured from the swollen fiber (Figure 19) varies from about 37 to 44 degrees. This is hardly in line with the parallelism implied in the literature on the cellulose orientation of flax and ramie—which is not surprising in that the fibers here pictured (Figures 16B, 18B, and 19) are swollen many times their natural

FIG. 12. *Forty-two-day-old fiber of Mexican Big Boll cotton, NO_2-treated 1 hour; swelled with 10 percent trimethylbenzyl ammonium hydroxide. Note solution of secondary wall to leave fragmentary fiber sheath. Magnification 250 ×.*

size, while the ceellulose orientation elsewhere reported was measured from x-ray diagrams of dried fibers. The effect of swelling on the arrangement of cell wall fibrils was noted by Hess [24], who points out that the x-ray angle for dry ramie fibers is 4–7 degrees, but the fibril angle in ramie fibers which have been swelled in cuprammonium hydroxide is 45 degrees. Unfortunately, some of the workers [33] in fiber structure seem to have ignored this change of angle due to swelling, and have implied a direct correlation between the x-ray diagram and the fibril orientation observed microscopically.

Discussion

In swelling studies on the microscopical features of fiber structure, pretreatment with nitrogen dioxide offers a new approach to investigations in this field. While the violence of the oxidation is obvious, it is also evident that the general relationship of cell wall elements is unaltered. At the same time certain of the pictures produced during the swelling of fibers after NO_2 treatment are at variance with some of the conceptions of cell wall structure recorded in the literature on unoxidized fibers.

In the light of nitrogen dioxide studies it is pertinent to ask whether the primary wall consists of more than one layer, or, rather, whether the "first layer of the secondary cellulose" does not belong to the primary rather than to the secondary wall, since it forms a part of the outside "fiber sheath" which peels off smoothly from the remainder of the fiber (Figures 9 and 10).

It might be as proper to call this layer the "inner layer of the primary wall" as to label it the "first layer of the secondary wall," for it differs from both, and, in mature fibers at least, is separated from the primary wall with great difficulty whereas it can be rather readily removed from the bulk of the secondary wall.

The occurrence of what appear to be reversals in the primary wall (as in Figure 8) might possibly be explained by inequalities in the swelling of the fiber, though similar configurations on the fibers in Figure 7 would indicate some structural foundation for this abnormality. The fact that the primary wall and inner sheath layers are inseparable in mature fibers makes it obvious that the apparent reversal in the primary wall is merely the rupture of the primary wall along the lines of the fibrils of which the inner sheath is composed, the primary wall reflecting in this instance any structural configurations brought about in the "winding layer" or inner sheath by the swelling treatment.

Further question as to the relationship of the inner sheath ("winding layer") and the outer sheath (primary wall) is raised by Figure 11b, in which the sheath appears to be made up of bands. It is possible that the cracks which form the edges of the bands are caused by the swollen spirals of the inner sheath lying just beneath the primary wall, but Figures 8 and 9 would indicate that the inner sheath has finer spirals than those edging the wide bands in Figure 11b. Although the drastic oxidation treatment may have caused considerable alteration to the fiber, the structures in Figure 11b are an indication of an original sheath structure of a definitely spiral nature.

In the work by Hock, Ramsay, and Harris [26]

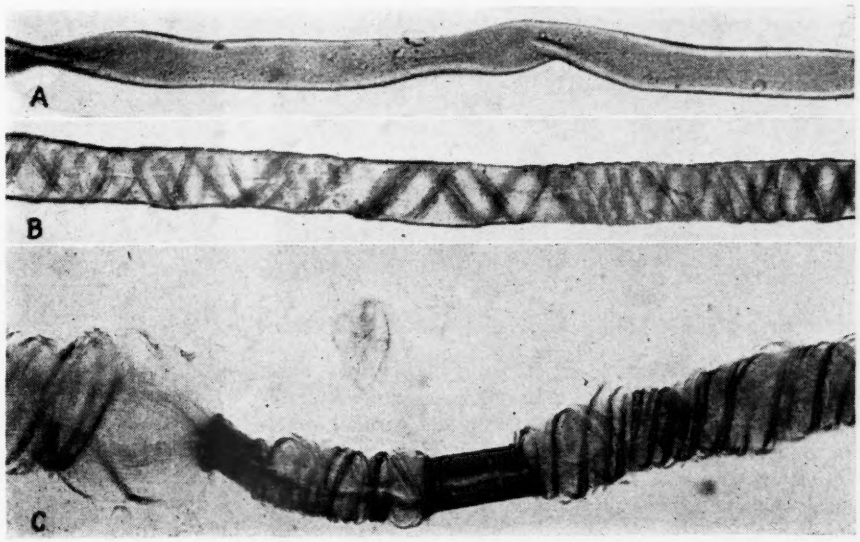

FIG. 13. *Progressive swelling of 42-day-old cotton fiber, NO_2-treated 1 hour. A—Unswelled. B—Swelled in dilute trimethylbenzyl ammonium hydroxide. C—Swelled in cuprammonium hydroxide. Note rupture of fiber sheath into spirals. Magnification 250 ×.*

polarized light has been used to show correlation in unoxidized fibers between alternating extinction bands along the length of the fiber with the occurrence of reversals in the thread-like fibrils which can be seen in a swollen fiber. If the reversal points in all (or at least an overwhelming majority) of the successive layers of the cell wall cellulose do not coincide, then the polarized light pattern is influenced not by the fibrillar arrangement but only by the varying thickness of the cellulose at any given point along the fiber. Since in the course of this study no spirals have been observed in the secondary wall of cotton fibers, no light is thrown by the NO_2 method on the occurrence or nonoccurrence of spiral fibrils in the porous areas of the lamellae, or the proportion of reversals occurring in immediate vertical succession at any given location on the length of the fiber.

In regard to the attempts to correlate the convolutions of the cotton fiber with the direction of reversal of the spiralling fibrils in the cell wall lamellae [26] it is difficult to follow this concept by microscopical observation. Only one or two layers of the total of 20 or 30 in any cotton fiber wall can be brought into focus at any given point on the fiber, and the fibrils are visible only after such drastic swelling that the convolutions have been removed.

There have been some attempts to correlate the angle of fibril orientation as observed microscopically with the crystalline orientation measured in x-ray diffraction patterns. However, x-ray diffraction patterns are usually made on bundles of untreated fibers, while photomicrographs of cell wall fibrils can be made only on drastically swollen single fibers. In none of the photomicrographs of cell walls used in the literature to illustrate this comparison of microscopical and x-ray orientation is it stated in which cell wall layer the fibrillar angle was measured, or to what degree the fiber has been swelled.

Although the spiral angle is described as the angle a fibril makes with the axis of the fiber, the angle indicated in the published photographs [5, 41, 42] is the angle one layer of fibrils makes with those of opposite direction in the layer immediately above or

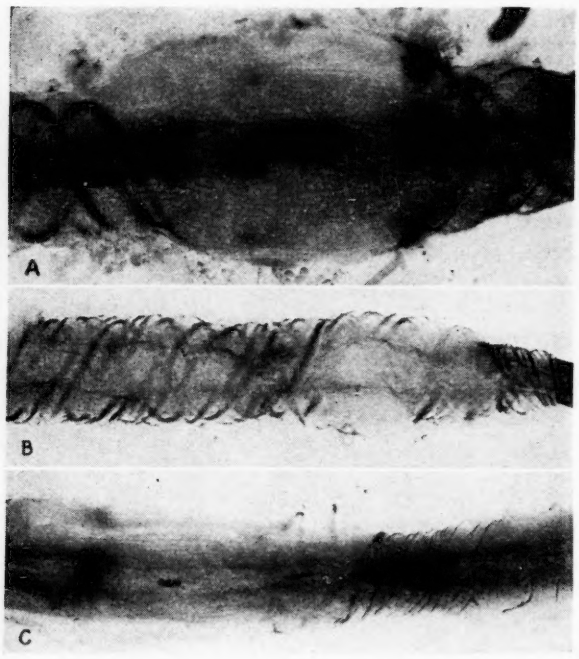

FIG. 14. *Progressive swelling of 42-day-old fiber, NO_2-treated 1 hour; swelled in cuprammonium hydroxide. A—Note spirals of fiber sheath and parallel lamellae of secondary wall. B—Note breaking off of fiber sheath to free secondary wall lamellae. C—Note separation and solution of secondary wall lamellae. Magnification 190 ×.*

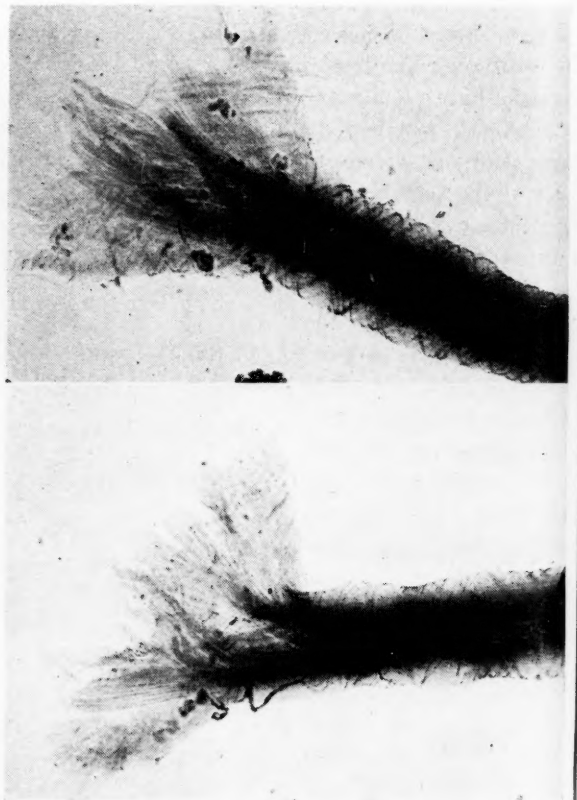

FIG. 15. *Swollen ends of broken 42-day-old fibers, NO_2-treated 1 hour; swelled in cuprammonium hydroxide. Note parallel lamellae of secondary wall; few remaining fragments of fiber sheath spirals. Magnification 200 ×.*

below it. This may or may not be the double-angle of the true spiral angle of either layer of fibrils because of the varying effect of swelling in successive layers, and because of the curvature of the fibrils in anastomosis with others of the same layer. While the authors [5, 33, 41, 42], no doubt, have meant to suggest only a general correlation between average microscopical fibril angle and average angle of x-ray orientation, their work has been interpreted by others to mean more than this, and some have gone so far as to suggest that by microscopical observation of the fibrillar angle some prediction could be made as to relative tensile strengths of different fibers; or even that changes in orientation within the fiber due to stretching could be detected microscopically. To date such evidence is extremely fragmentary.

Because of the difficulties inherent in the fineness of fibers it is doubtful whether conclusive answers to some questions on fiber structure can ever be obtained. It would indeed be helpful, however, in studies of heat-damaged, fungus-damaged, and chemically treated fibers to have a better picture of the typical cell wall against which to compare the effects of treatment. It is to be hoped that further use of the microscope in conjunction with chemical studies will serve to extend the frontiers already established by the voluminous and painstaking work devoted to this field. The use of nitrogen dioxide in this connection offers interesting possibilities.

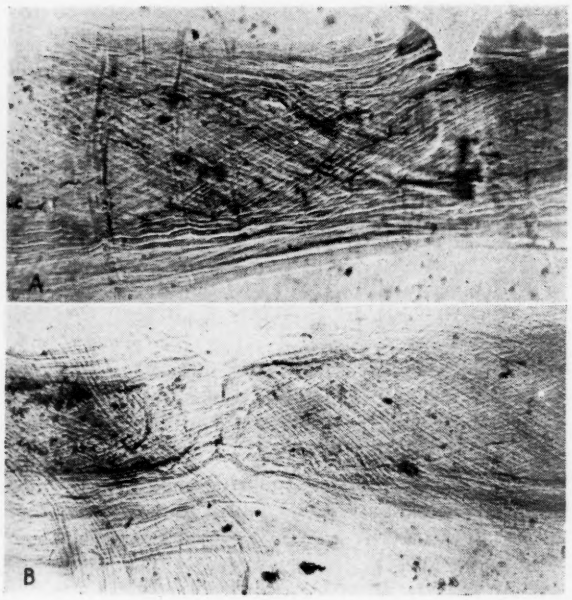

FIG. 16. *Ramie fibers, NO_2-treated 30 minutes; swelled in cuprammonium hydroxide. A—Fibril angle in secondary wall varies from approximately 27 degrees to 34 degrees with the fiber axis. B—Fibril angle in secondary wall varies from approximately 20 degrees to 34 degrees with the fiber axis. Magnification 75 ×.*

Summary

Nitrogen dioxide pretreatment is reported as a new technique for the microscopical study of fiber structure by swelling. Fibers which had been treated with nitrogen dioxide were observed microscopically during swelling with lyotropic reagents to study cell wall structure. Photomicrographs were made at various stages in the swelling process. Certain observations were made which seem to be at variance with those reported for unoxidized fibers. The differences noted may be due to the drastic effects of the NO_2 treatment or perhaps to the fact that mature fiber samples were observed in these studies, whereas most fiber studies reported in the literature are on fresh or living cotton fibers at various early stages of maturity.

1. The fiber sheath (primary wall and "winding layer") split into fine fibrils or into broad spiral bands, depending on the degree of NO_2-oxidation.

2. Reversals were observed in the fiber sheath; these may be in the "winding layer" or inner sheath beneath the primary wall, but adhered to it by the NO_2 treatment.

FIG. 17. *Milkweed bast fiber NO_2-treated 3 hours; swelled with concentrated NH_4OH. Note angle of fibrils beneath broken spiral of primary wall. Magnification 250 ×.*

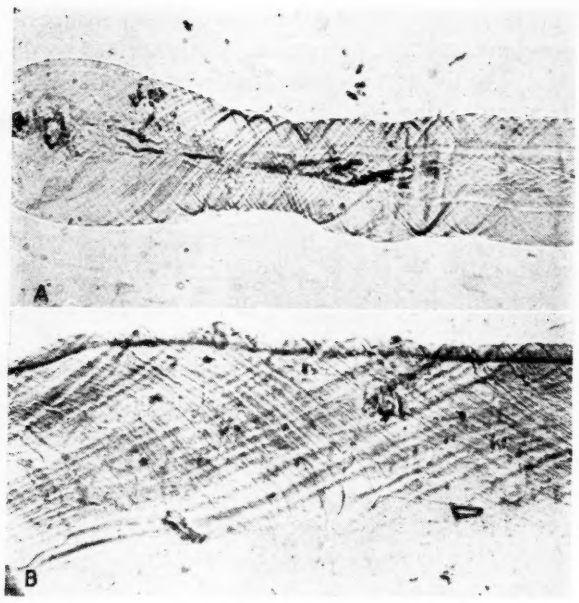

FIG. 18. *Ramie fibers, NO_2-treated 30 minutes; swelled in cuprammonium hydroxide. A—Fibril angle in primary wall varies from approximately 43–45 degrees with the fiber axis. B—Fibril angle in primary wall varies from approximately 28 to 45 degrees with the fiber axis. Magnification 200 ×.*

3. The fiber sheath appears to have a banded structure when not ruptured by swelling of the cellulose beneath it.

4. No spiral structure was observed in any layer of the secondary wall of cotton below the inner sheath, but the lamellae of the secondary wall seemed to disintegrate into fine fibrils which lie more or less parallel with the fiber axis.

5. Spiral fibrils were observed in the secondary wall layers of nitrogen-dioxide-treated ramie fibers and milkweed bast fibers; but the angle of their spiral orientation with respect to the fiber axis is several times greater than the slight angle of orientation seen in x-ray diffraction patterns. This is no doubt due to the extreme swelling to which the fiber has been subjected for microscopical study.

6. The spiral angle in the primary wall also varies with the degree of swelling, that for a ramie fiber varying from 28 to 45 degrees, and for a flax fiber from 37 to 44 degrees.

Acknowledgment

Acknowledgment is due Dr. R. E. Reeves and Mr. J. E. Giddens for preparation of the NO_2-treated samples of fibers. The author wishes also to thank

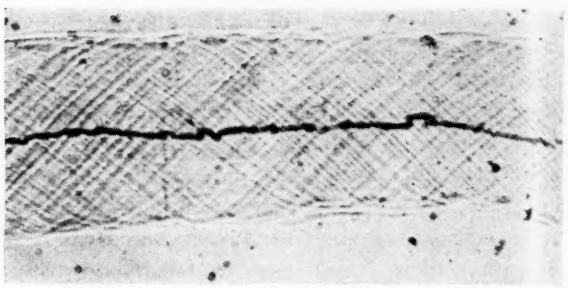

FIG. 19. *Flax fiber NO_2-treated 30 minutes; swelled with cuprammonium hydroxide. Fibril angle in primary wall varies from 37 to 44 degrees with the axis of the fiber. Magnification 200 ×.*

Miss Ines V. deGruy for her able assistance in the preparation of plates for this manuscript, and Mr. T. L. W. Bailey, Jr., for his helpful suggestions and criticisms. The kind criticisms of Drs. E. E. Berkley and Thomas Kerr of the Bureau of Plant Industry and Agricultural Engineering are gratefully acknowledged.

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Sizing of Spun Viscose Warp Yarns

V. Application of Corn Starch Ether Gum in Spun Viscose Warp Sizing and Its Removal from Fabric

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North Carolina State College School of Textiles

This is the last of a series of papers which describe certain parts of a program of research on warp sizing conducted by Textile Research Institute, Inc. Part I—"Effect of Stretch in Slashing," and Part II—"Abrasion Testing of Sized Warp Yarns," appeared in the January, 1945, issue of this JOURNAL. *Parts III and IV—"Application of Synthesize" and "Application of Methyl Cellulose," were presented in the February issue. A summary report of the entire program of research on the sizing of spun rayon warp yarns will be ready for distribution about May 1.*

EARLIER studies undertaken in the warp-sizing research have had as their principal objective the finding of a sizing agent or combination of agents which would qualify on as many as possible of the following:

1. Relatively high viscosity with a low concentration of solids. This requirement is considered necessary to permit building up a protective coat upon the surface of the warp yarn to prevent shedding of surface fiber without the use of an excessive amount of size.

2. Solubility in water. This property would facilitate ready removal of the size from the grey goods without the use of an enzyme and thus reduce the cost of the finishing operations.

3. Satisfactory adhesive qualities as reflected in the production of a sized warp with good resistance to surface abrasion and torsion.

4. Flexibility. The quality varies widely in various sizing materials and combinations of materials. The size should furnish protection without undue stiffening of the warp, as stiffness is accompanied by a high rate of end breakage through shearing of the warp at the heddles.

5. Extensibility. This quality is determined by measuring the elongation of the sized warp and has been found to affect greatly the weaving quality of the warp.

6. Stability. This requirement refers to stable viscosity of the agent in the form of size paste in the size box and in the form of size film on the warp and fabric.

With these objectives in view a product derived from corn starch ether gum on preliminary testing seemed to answer a great many of the requirements and was subjected to sizing and weaving tests involving the miniature slasher and full-scale slashing on the five-cylinder Cocker machine carrying a creel of eight section beams with 3,600 ends of 30/1 yarn spun from $1^{9}/_{16}$ inch $1\frac{1}{2}$ denier viscose fiber.

Summary

A corn starch ether gum was studied to determine its suitability for use in sizing spun viscose warps. The product was found to be soluble in cold water and the films formed from it were also soluble in cold water. Highly viscous size pastes were produced with a relatively low solids concentration. Satisfactory abrasion resistance and weaving quality resulted from use of the gum, but considerable shedding of fiber resulted in weaving. Fabric stiffness was observed to be approximately the same as that produced by corn gums with low solubilities.

Considerable advantage is indicated in finishing and dyeing because of the ease of removal of the size from the fabric. Complete removal of the size from the fabric could not be effected with cold water, but hot water and soap were found to produce satis-

* Research Consultant and Research Assistant respectively for Textile Research Institute's Warp-Sizing Project.

factory desizing. Direct dyes were used to dye the fabric in both a sized and a desized condition. It was found that satisfactory dyeing was possible with no preliminary desizing operation. Dyeing and desizing were effected simultaneously and the colors so produced were fully equal to those produced on fabrics desized by hot water and soap and by a bacterial enzyme.

Procedure

Properties of Corn Starch Ether Gum

Since this product is still in the development stages it has not yet been standardized. The particular lot of material used in this series of tests was prepared in the form of a solution containing 25 percent solids and having a pH of approximately 8 and a viscosity rating of 2,000 centipoises. The product was readily soluble in water and yielded a transparent film possessing the properties of flexibility, toughness, and complete solubility in water. In all preliminary tests and in the slashing operations the size maintained a high degree of stability. No tendency toward the formation of lumps in the size or scum on the surface of the size was observed.

Preliminary tests were conducted to determine the effect of temperature on the viscosity of solutions of various concentrations of the starch ether gum. The data from these tests are recorded in Table I and shown graphically in Figure 1. It should be observed that the solutions of higher concentration vary widely with changes in temperature, but in no case does coagulation occur as it did with the ether of cellulose described in Confidential Report No. 8. It will also be seen that sizing mixtures of extremely high viscosity are obtained by using a relatively small amount of solid material. Concentration No. V (3 pounds per gallon) was selected for the slashing and weaving tests. It should be observed that although 3 pounds of the concentrated solution were used in each gallon of size, there was only ¾ pound of solid materials per gallon of size since the concentrated solution contained only 25 percent solids. The data from Table I are presented graphically in Figure 2 to show the effect of concentration on viscosity, the temperature remaining constant for each series of concentrations.

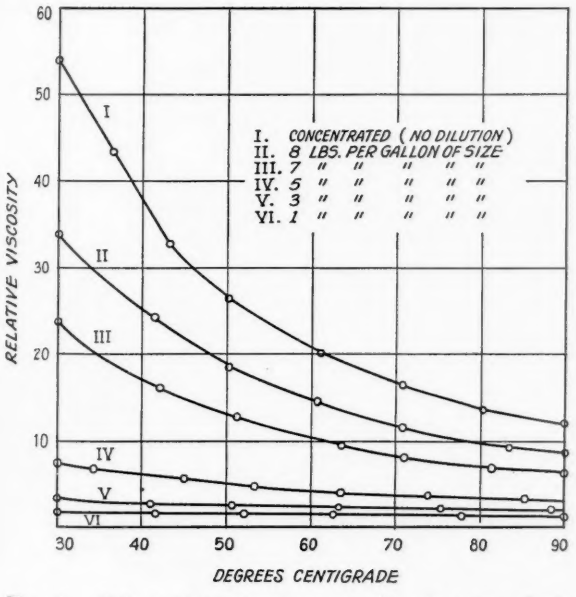

FIG. 1. *Effect of temperature on the viscosity of six solutions of corn starch ether gum.*

TABLE I. Effect of temperature on specific viscosity of 6 aqueous solutions of corn starch ether gum

Concentrated (no dilution)			5 lbs. per gallon		
Temperature, C°	Secs.	Relative viscosity	Temperature, C°	Secs.	Relative viscosity
27.5	207.3	57.6	27.0	28.0	7.8
36.5	155.3	43.1	34.5	23.3	6.5
43.0	117.3	32.6	45.0	20.1	5.6
50.0	95.5	26.5	53.0	15.8	4.4
61.0	72.0	20.0	63.5	13.9	3.9
70.5	59.3	16.5	74.5	12.4	3.4
80.0	47.7	13.3	85.0	11.1	3.1
90.0	40.4	11.2			
8 lbs. per gallon			3 lbs. per gallon		
30.0	121.5	33.7	30.0	11.1	3.1
41.5	86.4	24.0	41.0	8.9	2.5
50.0	65.3	18.1	50.5	8.5	2.4
60.5	51.9	14.4	63.0	7.9	2.2
70.5	41.1	11.4	75.0	7.1	1.9
83.0	32.8	9.0	88.0	6.8	1.9
90.0	29.9	8.3			
7 lbs. per gallon			1 lb. per gallon		
29.5	84.4	23.4	29.0	5.8	1.6
42.0	57.2	15.9	41.5	5.4	1.5
51.0	44.7	12.4	52.0	5.1	1.4
63.0	34.1	9.5	62.5	4.9	1.3
70.5	28.3	7.9	77.5	4.4	1.2
81.0	24.3	6.7	90.0	4.0	1.1
90.0	21.7	6.0			

Tensile and Abrasion Tests

The results of the tensile and abrasion tests are summarized in Table II. For comparison the data for methyl cellulose, an ether, and tapioca gum are also included. No significance is attached to the

TABLE II. Summary of tensile and abrasion tests

Sizing agent	Tensile strength	Residual elongation	Abrasion resistance	Ratio to unsized
	oz.			
Starch ether gum	8.48	10.61	41.9	5.25
Methyl cellulose	8.20	11.50	50.0	6.25
Tapioca gum	8.66	10.50	48.0	6.0

small differences in tensile strength and elongation. The warp was found to be slightly lower in abrasion resistance than the record for methyl cellulose and tapioca gum but was sufficient to permit weaving without fuzzing of the warp.

Size Distribution

Microscopic study of cross sections of the sized yarn indicated that considerable penetration had occurred. One of these sections was photographed (Figure 3). A slight tendency toward surface coating is evident. It should be observed at this point that shedding of surface fiber in weaving was high. The concentration was low, which yielded a size mixture of 1.9 (relative viscosity).

Fabric Stiffness

By using the stiffness indicator described in Confidential Report No. 8, a numerical stiffness rating may be set up for any size applied to the warp. This instrument is also useful in determining the degree of size removal accomplished by different processes of extraction effecting complete or partial removal of the size from the fabric. The instrument was used in this series of tests to determine not only the stiffness of the grey goods for comparison with fabrics containing other sizing materials but also for indicating the extent of size removal by several methods of extraction.

Records of these tests are given in Table III. The average rating in stiffness units for the grey goods woven from the warp sized with starch ether gum was 36.1, which is slightly higher than the rating obtained (35.5) with the use of a corn gum of the acid-converted type (Confidential Report No. 8, page 7), and considerably higher than that obtained with methyl cellulose (20.7).

Desizing and Finishing

It has previously been stated that the films formed from starch ether gum dissolve readily in cold water.

TABLE III. Stiffness ratings for grey goods and finished goods from warp sized with starch ether gum

Test No.	Grey goods*	Finished goods, desized with: Cold water	Hot water and soap	Enzyme (Rapidase)
1	35	21	12	10
2	37	20	11	10
3	34	21	12	11
4	37	19	12	10
5	38	22	11	10
6	37	21	12	10
7	35	22	12	10
8	35	21	11	9
9	36	21	11	9
10	37	22	12	11
Average:	36.1	21	11.6	10

* Ratings are in stiffness units. For explanation of method of measuring stiffness see Confidential Report No. 8.

To determine the type of finishing treatment necessary for fabrics sized with this product a number of desizing operations were carried out using cold water, hot water and soap, and an enzyme bath.

COLD WATER EXTRACTION. Samples of the grey goods were soaked for a period of 1 hour with frequent agitation to determine whether removal could be effected in this manner. After thorough rinsing, tentering, and drying, the fabric was noticeably stiffer than the completely desized fabric. Measurements indicated an average stiffness of 21, which is approximately double the rating of 10 recorded for the fully desized fabric. Therefore it is apparent that cold water alone does not completely remove

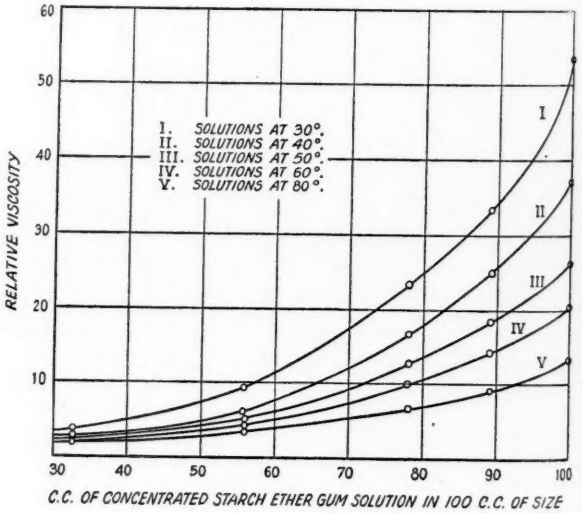

FIG. 2. *Effect of concentration on the viscosity of corn starch ether gum at various temperatures.*

the size from the fabric despite the high degree of solubility demonstrated by the film alone. This difference is accounted for by the high degree of penetration obtained in sizing the warp and the compact condition of the fibers within the fabric which tend to prevent or retard penetration by cold water.

EXTRACTION WITH HOT WATER AND SOAP. In the second treatment the fabric was steeped for 1 hour in a bath of hot water and soap (180°F). After rinsing, tentering, and drying, the goods exhibited a soft hand and had a stiffness of 11.6 units, which is only slightly above that (10) of completely desized fabric. The fabric finished in this manner gave a negative test with iodine solution. The conclusions were drawn that such a treatment can be satisfactorily used for both white and colored fabrics, but that a purer white may be obtained by the use of an enzyme.

ENZYME EXTRACTION. For enzyme extraction, Rapidase, a bacterial type enzyme, was used. The bath was made up with 3 percent of the enzyme and a small amount of common salt and the goods was steeped for 1 hour at 170°F. On tentering and drying, the fabric had a stiffness rating of 10. The fabric was whiter than that produced with warm water and soap.

Quantitative Size Extractions

The foregoing tests on size extraction may be regarded as qualitative since stiffness was the criterion used for indicating the amount of size in the fabric.

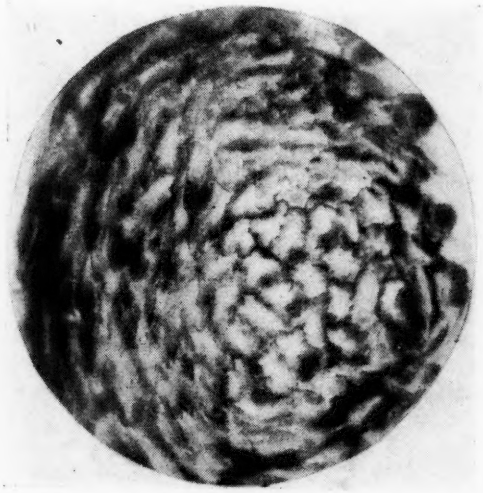

FIG. 3. *Photomicrograph of yarn sized with starch ether gum (magnified 400 ×).*

TABLE IV. Comparative solubilities of several sizing agents as indicated by the extent to which they may be removed from fabric with hot water and soap

Sizing agent	Percent size removed by: Enzyme	Hot water and soap	Percent of total size content removed by hot water and soap
Thick-boiling corn starch	4.8	1.0	21.0
Thin-boiling corn starch	5.0	2.2	44.0
Tapioca gum	5.2	3.4	65.4
Corn starch ether gum	4.9	4.4	90.0

For comparison three other fabrics containing approximately the same percentage of other available sizing agents were subjected to quantitative size extraction tests.

The data from these tests are recorded in Table IV. The sizing agents tested were thick-boiling corn starch, a thin-boiling corn starch of medium fluidity, tapioca gum, and corn starch ether gum. It was found that 90 percent of the starch ether gum yielded to the soap-and-water treatment. In the same period of time 65.4 percent of tapioca gum, 44 percent of thin-boiling corn starch, and 21 percent of thick-boiling corn starch were extracted. It is evident that the corn starch ether gum is considerably easier and simpler to extract than the other sizes tested.

Simultaneous Dyeing and Extraction

The most interesting observation made in this series of tests was the facility with which the grey goods may be dyed without desizing. Samples of the grey goods after wetting in warm water were entered in a dye bath containing: 2 percent Direct Blue RW (conc.), ½ percent Gardinol WA Dou. Powder, and 20 percent common salt.

The dyeing operation was continued for 1 hour and no unusual difficulty was experienced in securing penetration and level dyeing. Other direct colors have been used with similar results. The authors have thus concluded that grey goods containing starch ether gum size may be dyed with direct dyes with no preliminary boil-off or extraction. Dyeing and size removal are thus effected simultaneously in the same bath.

A Tumbling Device for Preparing Cellulosic Dispersions

S. A. Simon

A Contribution of the Research Division, Chicopee Manufacturing Corporation

In the method [1] ordinarily used by the textile industry in the preparation of cellulosic dispersions in cuprammonium or other solutions used in determining the cellulose chain length, the cellulosic specimen and the solvent are agitated by the repeated passage of a metal plunger along the length of a pipette viscometer containing a mixture of the two. This is usually accomplished by a rotating mechanism which gives the pipette an end-over-end tumbling action about an axis perpendicular to itself. This results in the plunger scraping one side of the pipette in one-half of the journey, and the opposite side during the return journey, leaving the remainder of the circumference of the pipette with adhering cellulosic material under certain conditions. If the adhering cellulose should happen to be in the path of the plunger, the reciprocating motion along the whole length of the pipette might be impeded. The effect of this incomplete agitation has been found to be sufficiently serious with celluloses of long chain length to cause unreliable results in perhaps 10 percent of the specimens run, and consequently the construction of a better type of tumbling apparatus was undertaken by the laboratories of the Chicopee Research Division.

In the design of the new tumbling device the usual end-over-end action was supplemented by a very slow rotation of the viscometer about its own axis in accordance with principles already suggested in the literature. In this way successive passes of the agitating plunger were distributed around the circumference of the viscometer rather than localized on two opposite sides only. With this axial rotation the plunger is enabled to seek out the weak spots in the adhering cellulose on the walls of the pipette and thus to initiate the removal and dispersion at an early stage. Continuation of this action clears the entire side wall, which might otherwise never be touched by the plunger.

This was accomplished in the apparatus shown in Figure 1, in which the main constructional features are visible. A heavy base casting supports a horizontal shaft which is driven at approximately 4.5 r.p.m. by an oil-immersed 80:1 worm reduction from the slow-speed shaft of a common commercial form of laboratory stirrer. The oil-immersed worm provides continuous lubrication for the worm and gear and the immersion trough catches any drip; the motor is provided on its high-speed shaft with a small fan and guard to prevent possible overheating on continuous operation.

The horizontal shaft of the mechanism carries two cylindrical cases, one at either end, in which a total of 24 viscometers can be placed and held securely in a cheesecloth packing. End covers with bayonet locks on the cylinders close the assembly and exclude light.

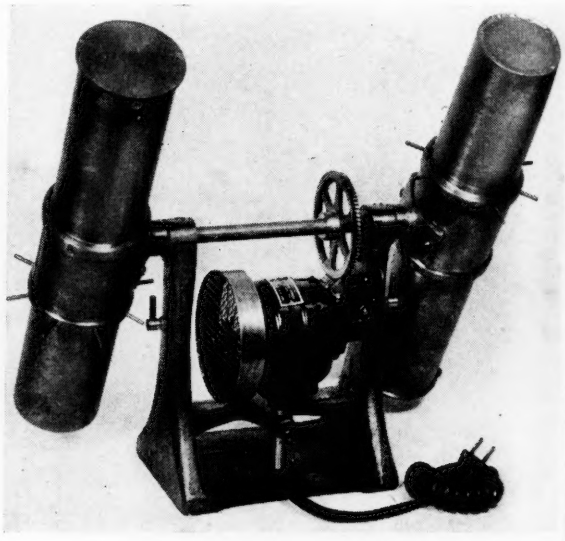

FIG. 1. *A tumbling device for preparing cellulosic dispersions.*

[1] Tentative Method of Test for Apparent Fluidity of Dispersions of Cellulose Fibers in Cuprammonium Hydroxide. A.S.T.M. Designation D 539–40T. *A.S.T.M. Standards on Textile Materials,* 1944, page 160.

The special rotation of the viscometers about their own axes is provided by rotating each cylinder in the mounting ring in which it is carried on the end of the horizontal shaft. This mounting ring merely holds the cylinder between fixed collars, and a friction spring guards against undesired rotation. Fixed to each cylinder is an eight-spoke star wheel made of projecting pins which engage a fixed pin, suitably located on the frame of the instrument, on each revolution about the horizontal axis. Through this arrangement each cylinder is rotated on its own axis, carrying the viscometers within it through the same motion, one-eighth revolution for each complete end-over-end cycle. The star wheel arrangement has the advantage of extreme simplicity and of practically complete freedom from jamming or entanglement with clothing, etc., which might occur in the usual differential mechanism. After a year's use the only attention required has been the oiling of the horizontal shaft and the mounting rings which carry the two cylinders.

This tumbling device has been used during the past year with greatly improved results. With the older type of apparatus, repeated tests sometimes failed to check even after an overnight period of tumbling, indicating incomplete dispersion. To get sufficient precision in the results it was often necessary to run four separate specimens to get an average. With the new device the overnight tumbling period seems entirely adequate and the sensitivity and accuracy seem much improved. While check tests are still run to guard against accidental errors, a single test seems to be sufficiently precise for present requirements. It might be noted that this apparatus satisfies essentially A.S.T.M. requirements, and since it can be designed for any required end-over-end tumbling speed and axial rotation it is a possible basic apparatus for specification purposes.

Acknowledgment

The author wishes to acknowledge the assistance of Mr. John T. Frydryk, mechanician of the Research Division of the Chicopee Manufacturing Corporation, who designed and constructed this device from the suggestions of the chemistry staff, and Miss Alta King, secretary of the Research Division, who typed the manuscript.

Spun Rayon Warp Sizing Summary Report of Institute Project

FOLLOWING the conclusion of a series of practical experiments on warp sizing of *filament* rayon, results of which have been published previously, Textile Research Institute, Inc., undertook a systematic study of the weaving efficiency of *spun* rayon yarn and of the relationship between the weaving efficiency and the film properties of warp sizing materials.

The results of this study were reported to the sponsors of the projects in a series of fourteen confidential reports. A summary of the results now has been prepared for publication, and it is expected to be ready for distribution early in May.

The first part of the report covers tests conducted at the North Carolina State College School of Textiles, and the second part deals with the concluding phases of the study conducted as a joint project of the Institute and of the Textile Foundation, in which slashing and weaving experiments and yarn tests were performed at the Textile School, and film studies were made at the Textile Foundation laboratories, National Bureau of Standards.

Copies of this report will be distributed without charge to company members of the Institute. Others may secure it from the Institute at a cost of $1.50 by writing to the Textile Research Institute, Inc., 10 East 40th Street, New York 16, New York.

ABSTRACTS

SERVICES AVAILABLE

Reprints of the original articles abstracted in TEXTILE RESEARCH JOURNAL *are often available from the publishers. Otherwise, it may be possible to secure copies of the periodicals. A list of periodicals, the abbreviations used in references to articles, and the addresses of publishers of English language publications was given on page 22 of the January, 1945, issue. Orders should be sent direct to the publishers.*

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ANALYSIS: TESTING LABORATORY METHODS

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Antiseptic Fabrics

A suggested method for thorough testing of antiseptic fabrics. L. C. Barail. *Am. Dyestuff Reptr.* **33**, P529–36 (Dec. 4, 1944).

Antiseptics of the phenyl mercury group are the only ones which have been found to meet the requirements of very low toxicity toward humans and resistance to multiple washings as well as high performance, lack of odor, and lack of color, when applied to fabrics. Within the group there is great variation in efficiency and toxicity. To meet the present need for evaluation procedure for fabrics treated to render them antiseptic a complete and systematic method for testing such fabrics is suggested. The plan consists of two groups of tests, the first of which is considered informative and includes: (1) toxicity—minimum lethal dose test; (2) skin irritants—(a) animal injection test, (b) patch tests; (3) sensitizers—patch tests, (4) antiseptic value—determination of phenol coefficient. The second test group is for performance and the tests are conducted on fabrics as is and after, up to 30 washings. This group consists of tests for (1) sterility; (2) bacteriostasis against (a) *Staphylococcus aureus*, (b) *Eberthella typhosa*, (c) *Escherichia coli*; (3) bactericidal value after contamination with E. coli; (4) fungistasis against *Trichophyton gypseum*, and (5) fungicidal value against *T. gypseum*. Details of all of these tests as performed on four members of the phenyl mercury group (applied to cotton-wool, and to all cotton fabric) are described and the results interpreted; 25 tables summarizing the individual tests are included. Fabrics containing wool are more resistant to washing when treated with this type of compound, apparently because of better original penetration. It is noted that one phenyl mercury compound was outstanding; it passed every test after 25 washings. No compound has been found within the limits of toxicity which will pass all tests after 30 washings.

Cellulose Analysis

The use of Br in the estimation of cellulose in plant materials. J. G. Shrikhande. *Biochem. J.* **38**, 207–9 (1944) (through *Chem. Abstr.*, 1944, **39**, 615[4]).

Material should be uniformly ground (64–80 mesh). Stir 1–2 g. into 100 ml. 3% Na_2SO_3, bring to a boil and filter. Transfer the washed material to 100 ml. H_2O in a beaker, add 10 ml. bromide-bromate mixture (dissolve 2.5 g. $KBrO_3$ in 10% KBr to make 100 ml. solution), then 5 ml. 10% (w./v.) H_2SO_4 and allow to stand 15 minutes with stirring. Filter with suction, wash with H_2O, and transfer the material to a beaker containing 50 ml. H_2O and 50 ml. 6% Na_2SO_3. Keep for 20 minutes on a boiling water bath. The bromination must be repeated several times until only a faint purple color remains. Suspend the cellulose in 100 ml. H_2O, add 10 ml. bromide-bromate mixture, 10 ml. H_2SO_4, and, after development of the reaction, about 5 ml. 10% NaOH until the liquor is colorless. After washing, extract the material again with 100 ml. 3% Na_2SO_3, and remove the sulfite by boiling for 2 minutes with 250 ml. H_2O, filter in a Gooch crucible, dry, and weigh. The powdered material is used for furfural and lignin determinations. The advantages of the method in the tropics are discussed.

Fiber Identification

Identification of new fibers and chemicals in rag cuttings. F. L. Simons. *Paper Mill News* 67, 14, 16, 18 (Dec. 16, 1944) (through *Bull. Inst. of Paper Chem.* **15**, 159, Jan. 1945).

Reference is made to the difficulties introduced by the various synthetic fibers, waterproofing finishes, and similar treatments of rags for papermaking. The rag room foreman needs a test which will tell him whether a given rag is cotton or not, even if it does not actually identify the fiber, if it is not cotton. Based on the procedure of the American Association of Textile Colorists and Chemists in their 1944 yearbook, a simplified scheme of analysis is suggested, involving two tests—the effect of the flame and solubility in a solution of phenol in acetone. Any fiber or fabric that burns with the formation of a melted lump or simply melts without burning or is wholly unaffected by the flame, is almost certainly unfit for papermaking. If, because of blended yarns, the flame test is inconclusive, any fiber or fabric that dissolves or softens in the acetone-phenol reagent is probably unfit for papermaking. For identifying the regenerated cellulose fibers (viscose and cuprammonium), the author recommends staining with Texchrome obtainable from Eimer and Amend, in preference to Brilliant Blue 6BA used in the AATCC method. The identification of special waterproofing or other chemical treatments is far more complex than the identification of fibers. These treatments vary so widely that actual identification is almost impossible. By and large, it is a matter of the alertness and experience of the personnel in the rag room to spot these special finishes by their appearance or handle. At the present, nothing of definite usefulness for the rag problem with regard to identification procedures exists. A chart outlining the tests recommended by the AATCC for identifying synthetic fibers is included. In conclusion, some of the details of the tests as used in the rag room and laboratory of Crane and Company are outlined by M. E. Lynch.

Hydrostatic Pressure Tests

A comparison of the AATCC and the Suter hydrostatic pressure testers. G. J. Mandikos and B. A. Ryberg. *Am. Dyestuff Reptr.* **34**, P37–40 (Jan. 15, 1945).

Probably because of the smaller area subjected to test, the AATCC Hydrostatic Pressure Tester gives a higher reading than the Suter Tester, which operates on the same principal. Tests were conducted to determine what mathematical relationship if any exists between these two methods, which are the most widely used for measuring the resistance of fabric against a column of water. Ten samples of 20 fabrics were used in each tester at three temperatures, 70°, 80°, and 90° F, for a total of 1,200 tests. A straight line relationship was found to exist between the values obtained by the two testers corresponding to $A = 1.32S - 4$ where A is the reading in centimeters on the AATCC tester and S the reading on the Suter Tester. Temperature variation between 70° F, and 90° F had no appreciable effect upon this relationship.

Polymers and Copolymers

The behavior of polymers and copolymers. R. F. Boyer. *Can. Chem. and Proc. Ind.* **28**, 528–30 (Aug. 1944).

If one measures the volume or length of a plastic as a function of temperature, two straight lines of different slopes are obtained. The point of intersection defines a temperature that is characteristic for each plastic, and which has been given various names, such as second order transition point, brittle point, etc. In effect it constitutes a fairly precise measurement of the internal melting or softening point of a high polymer. This transition temperature correlates quite well with other mechanical properties such as cold flow, low temperature, brittleness, electrical behavior, moisture, vapor transmission. Illustrative data are given for styrene-butadiene copolymers and vinyl chloride-vinylidene chloride copolymers.

Polyvinyl Chlorides

Some rheological properties of polyvinyl chlorides. L. Bilmes. *J. Soc. Chem. Ind.* **63**, 182–5 (June, 1944).

The rheological properties of plasticized polyvinyl chloride have been studied in a specially constructed torsion apparatus over the temperature range 70° to 100° F. The results obtained have been analyzed by means of the Nutting equation and their significance is related to considerations of molecular structure. As the temperature is raised from −70° to +140° F the material passes through four states—the glassy, leathery, rubbery, and plastic states—and it has been found possible to characterize these states by values of the constants of the Nutting equation.

Analysis of Preventol GD

A new method for the analysis of preventol GD in vat and mineral dyed textiles. D. A. Shiraeff. *Am. Dyestuff Reptr.* **34**, P15–16 (Jan. 1, 1945).

A convenient method has been developed for the determination of the mildew-proofing agent 2,2′-dihydroxy-5,5′ dichloro-diphenyl-methane (Preventol GD) in textiles. The existing official method determines the organically bound chlorine, whereas the new method depends upon the color reaction of phenolic compounds with bleaching powder, and is much simpler and shorter. The material to be analyzed is extracted with an alkaline solution of sodium hypochlorite and the intense yellow color of the extract is compared with known standards prepared by treating identical fabric containing no Preventol GD with known amounts of this product and then treating in the same way as the unknown sample. A method is also described which can be used if no untreated, otherwise identical material is available.

Protein Analysis

Qualitative analysis of proteins: a partition chromatographic method using paper. R. Consden, A. H. Gordon, and A. J. P. Martin. *J. Biol. Chem.* **38**, 224–32 (1944) (through *Bull. Inst. Paper Chem.*, **15**, 136, Dec. 1944).

In the present paper, a qualitative micro-analytical method for separating amino acids on partition chromatograms is described, using cellulose in the form of strips of filter paper as solid phase, and phenol, collidine, and *n*-butanol as mobile phases. Ninhydrin is used to reveal the amino acids. Normal water content of the paper being assumed, the calculated partition coefficients are close to those directly measured, showing that the cellulose acts as an inert support. Two-dimensional chromatograms on sheets of filter paper are described; first, one solvent is run in one direction; then, after the paper has been dried, another solvent is run in a direction at right angles to the first.

Reinforced Synthetic Resins

Microscopic examination of reinforced synthetic resins. Clementina M. Gordon. *J. Soc. Chem. Ind.* **63**, 272–7 (Sept. 1944).

A technique is described for preparing microscopic sections of plastics and for differentially staining the various kinds of resin and fibers intended for use as structural materials. There is evidence that phenolic resins can penetrate the cellulose fiber wall and, on condensing there, form a combination, possibly of the lignin-cellulose type, which alters the staining and optical properties of the fiber. This does not occur at precure, but subsequently in the press, owing to the temperature and not to the pressure. It is much modified by the different kinds of fibers, and slightly by the kind of resin; the strength of the resin-fiber bond increases with this modification of the fiber, and influences the way in which the board fails and its property of pseudo-ductility. The microscopic examination of a board gives no quantitative information about its mechanical behavior, but from a qualitative point of view plastic microscopy might possibly become a practical adjunct to mechanical testing.

Starch Fluidities

Starch fluidities. Piedmont Section, AATCC. *Am. Dyestuff Reptr.* **33**, P513–18 (Dec. 4, 1944).

The need for a standardization of procedure and nomenclature with reference to starch fluidities is emphasized. The fluidity number under which a given starch is sold is at present meaningless to the consumer unless he is familiar with the method which has been used to determine it. Three methods now in use for the determination of starch fluidities are presented in detail. Results of tests are described which show how two starches of the same fluidity number may differ widely in actual fluidity. Different laboratories using the same method of test on the same starches showed considerable variation in result. Single samples may be accorded widely divergent fluidities when different methods are employed. Under controlled conditions on the same apparatus different operators can obtain quantitative checks.

Measurement of Surface Tension

An apparatus for the determination of surface and interfacial tension. Otto F. Steinbach. *J. Chem. Ed.* **21**, 582, 601 (Dec. 1944).

Apparatus is described for the measurement of surface tension which employs both the capillary rise and bubble pressure methods alternately, using the same sample for each determination. Results by the two methods agree within 5%.

Viscosity Determination

The rheological concepts of viscosity and yield value. Henry Green. *Interchem. Rev.* **3**, 105–111 (1944).

The differences in properties of various common substances are described as an aid in visualizing the flow properties measurable as viscosity (for simple liquids), plastic viscosity, and yield value (for solid-liquid dispersions). Principles of rotational viscometers of the Stormer and MacMichael types are described, and the equations which are used to measure viscosity and yield value in absolute units are given. The author emphasizes the necessity for determining rates of flow at several shearing stresses in order to obtain a complete flow curve for a plastic material; inadequacy of a single-point measurement is shown. The following terms are defined: shortness, length, consistency, body, thixotropy, tack, coefficient of viscosity, yield value.

Wax in Cotton Fiber

Determination of wax in cotton fiber. Carl M. Conrad. *Ind. Eng. Chem. Anal. Ed.* **16**, 745–8 (Dec. 1944).

A new technique is described for the determination of the wax of cotton fiber. It consists of a two-step process in which the wax is first extracted with hot 95% ethyl alcohol and then transferred to chloroform through a phase-separation process, in order to eliminate sugars, mineral constituents, and other non-waxy constituents removed at the same time by the alcohol. The wax is extracted from cotton fiber more rapidly by hot alcohol than by chloroform, the most rapid and adequate of a considerable number of common wax solvents previously studied. The wax thus determined contains a negligible amount of mineral impurities and is less contaminated with sugars than that determined in the usual Soxhlet extraction.

CHEMICAL AND PHYSICAL RESEARCH

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Contraction Phenomenon

A reversible contraction phenomenon in animal hairs. C. S.

Whewell and H. J. Woods. *Nature* **154**, 546 (1944) (through *Chem. Abstr.*, 1944, **39**, 623[6]).

If a wool fiber is immersed in cuprammonium hydroxide solution it becomes stained greenish blue and contracts in length as much as 28% of the original length. If the fiber is then placed in dilute H_2SO_4, the blue stain disappears and the fiber returns practically to its original length. The dimensional changes are accompanied by changes in the x-ray photograph.

Color Standards

Colorimeters and color standards, O.S.A. Committee on Colorimetry. *J. Opt. Soc. Am.* **35**, 1–25 (1945).

This is Chapter VIII of the "Report of the Colorimetry Committee of the Optical Society of America." This report may be considered the outstanding work on this subject. It discusses various types of colorimeters, both visual and photoelectric; transparent color standards; color standards of reflecting surfaces, including descriptions of the Munsell system and the Ostwald. It discusses color dictionaries, including Ridgway and Maerz and Paul, also special color standards such as those of the Textile Color Card Association of the United States. The chapter concludes with references to the ISCC-NBS color names and to the American Emergency Standard for Colorimetry adopted by the American Standards Association in 1942.

Hydrolysis of Cellulose

The rate of hydrolysis of cellulose. Preliminary communication. T. Sihtola. *Svensk Kem. Tid.* **55**: 177 (July, 1943); *Chem. Zentr.* **114**, II, 1802 (Nov. 17, 1943). (Abstract in German only available) (through *Bull. Inst. of Paper Chem.*, **15**, 155, Jan. 1945).

The hydrolysis was carried out in concentrated aqueous zinc chloride solution, the H-ion activity of which was determined by following the rate of hydrolysis of sucrose. That part of the cellulose hydrolysis which could still be followed viscometrically required the same time as did the complete inversion of sucrose. The temperature, however, was 30° higher than in the case of sucrose.

Oxidation of Cellulose

Oxidative degradation of polysaccharides. Georg Jayme, Martin Satre, and Spyros Maris. *Naturwissenschaften* **29**, 768–9 (1941); *Chem. Abstr.*, 1944, **38**, 2197[1,8]. (In German; abstract in English. Abstract only available) (through *Bull. Inst. of Paper Chem.*, **15**, 155, Jan. 1945).

Low yields of glyoxal and erythronic acid were obtained when cellulose or starch was oxidized with 10% periodic acid at pH 1. Oxidation of cellulose with buffered 10% periodic acid at pH 3.5–4.0 transformed C_2 and C_3 into aldehyde groups in 97.5–99% yield, the polymer otherwise remaining unchanged. The new compound was insoluble in the oxidizing solution, but 95% was soluble in boiling water. After hydrolysis in 0.1 *N* sulfuric acid, glyoxal in 39.3% yield could be isolated as the phenylosazone. Xylan, oxidized and treated in this manner, yielded 62.4% of glyoxal. Glyceraldehyde, formed during the oxidation from C_3, C_4 and C_5 of the carbohydrate, was isolated in 44.9% as the methylglyoxalosazone by distillation from 20% sulfuric acid. Further oxidation of the original periodic acid oxidation product gave rise to glyceric acid which was isolated after sulfuric acid hydrolysis in 65.2% yield as the brucine salt. When the original periodic acid oxidation product was reduced with hydrogen and a Raney-nickel catalyst, glycerol and glycolaldehyde were isolated after hydrolysis. Better yields of the latter two compounds were obtained by frequently alternating reduction and hydrolysis. In this manner, a mixture of glycerol and glycol was obtained in 51.6% yield, 76.5% of which was glycerol. Oxidation of powdered straw with buffered periodic acid yielded 20% of a soluble oxidation product. The insoluble material upon further bromine oxidation gave rise to a mixture of organic acids.

Fiber Structure

The structure of plant-cell walls. VII. The question of the location where cellulose is formed in the hair cells of cotton. W. Wergin. *Planta* **32**: 535–46 (1942); *Chem. Abstr.*, 1944, **38**, 2361. (Abstract only available) (through *Bull. Inst. of Paper Chem.*, 15, 160, Jan. 1945).

The colorless particles that appear in the cytoplasm of cottonseed hairs are not cellulose structures from which the fibrils are formed by linear extension as stated by Farr (*cf. B.I.P.C.* **12**: 139–40), but plastids in which by special treatment starch can be demonstrated. Microscopy also shows that these plastids are not in contact with the fibrils as if the better to be incorporated into the cell walls; it is more likely that the formation of cellulose takes place at the walls.

Structure of the walls of phloem fibers. R. D. Preston. *Chronica Botan.* **7**, 414 (1943); *Nature* **154**, 366 (Sept. 16, 1944) (through *Bull. Inst. Paper Chem.*, **15**, 127, Dec. 1944).

Because of their commercial value, the fibers of the phloem (sclerenchyma) have so far been chiefly studied; in these, the x-ray diagram indicates the presence of cellulose chains in the longitudinal direction only, whereas observations on swollen walls by optical methods have led to the view that at least two layers are present and that they differ in their directions of the cellulose chains. Crossed cellulose chains definitely occur in the walls of certain algae. The x-ray diagrams of fibers of hemp and jute reveal the presence of cellulose chains in one direction only, running parallel with the major extinction plane; this diagram remains the same for fibers of different degrees of wall thickening, suggesting homogeneity of wall construction. However, by optical examination of swollen walls in

cross-section, there is indication of heterogeneity, which does not appear to be accounted for entirely by differential distribution of lignin and pectin. Differential swelling of the walls in different regions leads to the production of striations of various kinds. Also, the swollen material is easily broken into separate fibrils with associated change in direction of cellulose chains, which appears to have misled at least one worker. Swelling of the outer wall produces a "ballooning" of the outer wall layer in hemp, but not in jute, and this fact, associated with observed optical phenomena, suggests that the outer layer in hemp and the inner layer in jute differ appreciably from the rest of the wall. It seems clear that, in such walls, the aggregates of the cellulose complex must differ in their association with one another in the different layers. Comparisons with long collenchyma cells suggest that the optical heterogeneity may be caused by an angular dispersion of the cellulose chains from layer to layer; this argument is less convincing for hemp and jute, but not precluded by the x-ray diagram. There is, therefore, still doubt as to whether any chains exist in the secondary wall of the phloem fibers other than those which run in the longitudinal direction.

The importance and recognition of the supermolecular structure of cellulose hydrate fibers. H. Siebourg. *Zellwolle, Kunstseide, Seide* **46**, 215–23 (1941); *Chem. Abstr.*, 1944, **38**, 2198. (In German; abstract in English. Abstract only available) (through *Bull. Inst. of Paper Chem.*, **15**, 160, Jan. 1945).

The properties of artificial fibers are to be attributed not only to the degree of polymerization of the original cellulose and the x-ray orientation, but also to their internal structure. In order to gain some insight into the principles by which the various fibers are built up. Studies were made of the rates of acetylation and solution of such fibers by treating them for about 10 minutes with a 1:1 mixture of acetic acid and acetic anhydride after preliminary soaking. Examination of the cross-sections of the fibers under the microscope revealed that, in most viscose rayons, the interior dissolved first, after which the surrounding external layers disintegrated. When an insufficient amount of anhydride was used, only a very marked swelling was observed. Since it is impossible to reproduce the rate of solution of the fiber to the second, it is recommended that a reference fiber previously dyed with Korinth RK or Brilliant violet BBK be introduced with each test. Comparison of two fibers of the same origin showed that the fiber which was more resistant to wetting also withstood solution longer. With Schwarza and Lanusa fibers, the solution process attacked all portions of the fiber cross-section simultaneously. Cuprama fiber behaved in a similar manner. Comparison of viscose fibers from 5, 7, and 10% solutions showed that fibers spun from the most dilute viscose dissolved first; fibers from the 7 and 10% solutions showed no difference in this respect. Of fibers stretched to various degrees, those which were stretched least dissolved first.

Kehlheim Hollow Fiber

I. Development, preparation, fiber properties. R. Stoll. *Zellwolle u. Kunstseide* **2**, 51–61 (1944) (through *Chem. Abstr.*, **39**, 1945, 419[9]).

The hollow space is formed by generation of gas during coagulation. The details of preparation are not given, but it is claimed to be totally different from any similar product, yielding a fiber with better physical properties and greater uniformity. The specific volume of one type of Kehlheim hollow fiber is 2.50 cc./g. as compared to 1.40 for normal staple rayon. The thin-walled fiber collapses into a flat, cottonlike, ribbon. The inside walls of the lumen never seal up, however. Swelling in water is increased 20–30% and drying time is increased 15–25%. Dye absorption is greater than for other viscose fibers. With the darker dyestuffs, and especially after ironing, there is an undesirable glossy appearance to the fibers which cannot be overcome by the addition of dulling pigments.

Starch

Digestion of raw starch. A. K. Balls and Sigmund Schwimmer. *J. Biol. Chem.* **156**, 203–10 (1 plate) (Nov. 1944) (through *Bull. Inst. Paper Chem.*, **15**, 144, Dec. 1944).

Uncooked starch may be readily and completely digested by a mixture of extracts of hog pancreas and Aspergillus aryzae (mold bran). The breakdown of the starch granules may be observed without difficulty under the microscope. Both the pancreas and mold preparations contain factors (presumably enzymes) that are thermolabile. An inorganic factor present in the ash of wheat flour is necessary for rapid digestion. Calcium chloride may be used instead of this ash. Approximately all of the starch is converted to sugars. The products of the digestion are glucose and maltose, the former predominating. The mold bran is the source of maltase. The optimum pH of the digestion is about 5.2. Temperatures up to 55° are permissible with the quantities of enzyme reported here. Different starches vary in the readiness with which they are broken down. Potato starch is relatively resistant compared with corn and wheat starches. The size of the starch grains does not seem to be a determining factor in this resistance. 14 references.

Synthesis of amylopectin. W. N. Haworth, S. Peat, and E. J. Bourne. *Nature* **154**, 236 (Aug. 19, 1944) (through *Bull. Inst. Paper Chem.*, **15**, 144, Dec. 1944).

The authors report the isolation of an enzyme system from the potato which catalyzes the conversion of glucose-1-phosphate into a polysaccharide resembling the amylopectin constituent of natural starch. The product consists entirely of *d*-glucose units, gives a purple-red

color with iodine, is soluble in water, and does not retrograde from solution. Under identical conditions, synthetic and natural amylopectins are converted to maltose by β-amylase to the extent of 45 and 49%, respectively. Methylation data indicate quite definitely that the substance is not amylose. It is believed that Hanes' amylose synthesizing enzyme (*cf. B.I.P.C.* **10**: 309–10) participates in the reaction along with another factor—the Q factor—which enables 1,6-glucosidic linkages to form in addition to the normal 1,4-glucosidic bonds.

Starch Viscosity

Effect of electrolytes on the viscosity of solutions of starch paste. Augustin Boutaric and Madeleine Chapeaux. *Compt. rend.* **214**, 949–50 (1942) (through *Chem. Abstr.*, 1945, **39**, 236[9]).

The viscosity of the solutions of starch paste proved to be analogous to those obtained for other colloidal suspensions, such as bentonite, agar-agar, gum arabic, colloidal S and As. Upon increasing the concentration of electrolyte, the viscosity at first decreases, passes through a minimum, and thereafter increases. The concentration for which the minimum viscosity is observed and the value of this minimum are determined by the valence of the cation for electrolytes of the same anion.

Viscose Process—Effect on Fiber Strength

Changes in strength of single cotton fibers during the course of the viscose process. H. Hoffmann. *Zellwolle u. Kunstseide* **2**, 45–8 (1944) (through *Chem. Abstr.*, 1945, **39**, 413[1]).

It was shown that all the chemical operations of the viscose process, carried out so as to retain the fiber structure, exert a surprisingly slight effect on the strength of single cotton fibers. Dry strength is hardly diminished provided the ripening period is not extended extremely long. A longer period of ripening causes lowering of wet strength. However, the lowest relative wet strength was still 86% and thus much higher than the 50% of rayon. The decided lowering of fiber quality between cotton and rayon thus takes place only after the solution process. This may suggest the possibility of avoiding the completely dissolved stage as by utilizing directly the plastic properties of the xanthate.

BLEACHING: DYEING FINISHING

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Coir Dyeing

Coir dyeing. K. Kylas. *Indian Textile J.* **54**, 459 (1944) (through *Chem. Abstr.*, 1944, 39, 623[2]).

Coir is the fiber extracted from the fibrous hides of ripe coconuts. Its lightness, elasticity, and tensile strength make it excellent material for furnishing articles such as mats and matting. Dyes of the direct group have good affinity for coir but lack the economy of the covering and brightness of equal proportions of basic dyes. Acid colors have good affinity for coir and are fast to light. Members of the basic group have direct affinity for coir but are not fast to light. The coir can be entered in the dye bath without a preliminary wetting out and the dyeing started in the cold. When successive lots are dyed in the same shade, to economize on the cost of dyeing the same dye is used over and over. When dyeing a mixture of basic dyes, it is best to start with a fresh bath to obtain uniform shades in repeat dyeings. Basic dyes giving good results in coir dyeing are listed.

Dyeing Synthetic Fibers

A survey of the dyeing of the synthetic fibers. A. W. Etchells. *Am. Dyestuff Reptr.* **34**, P29–P32 (Jan. 15, 1945).

Frequent manufacturing changes in synthetic fiber production resulting in improved properties reflect also in changed affinity for dyes. Stretching procedures which produce a high degee of orientation with a consequent strength increase sometimes lack complete uniformity. These and other factors constitute a major problem for the dyer. The general dyeing properties and new developments in the dyeing of the important synthetic fibers are discussed. Efforts to overcome gas fading in dyed acetate goods by careful selection of dyes and by use of new methods, such as dyeing with acid dyes in the presence of organic swelling agents, have been only partially successful. In the latter case processing requires rigid control; inhibitors are of limited life and are removed by washing or cleaning. Nylon is best dyed with acetate dyes which give level dyeings of good light fastness but of only fair washing fastness; other types of dye are satisfactory only in a few instances. A new procedure on nylon which has merit consists of padding with certain chrome colors plus shellac, drying, ageing, and finally chroming at the boil. Vinyon, which is infrequently dyed, requires a swelling agent in the dyebath and is colored by acetate dyes, with rather poor light and wash fastness; the fabric is rendered useless by temperatures in excess of 150° F; Vinyon E, however, can withstand high temperatures. Problems with Aralac arise from its low wet strength, its sensitivity to alkali, and great affinity for most types of dyes; fair whites may be obtained on Aralac in combinations by treating with sodium hydrosulfite, then tinting with acid violet; chlorine or peroxides cannot be employed. Fortisan shows little affinity for most colors, four to six times as much dye being required as for a given shade on viscose rayon. Certain developed colors give the best results. Because saponification during manufacture may be uneven a pretreatment with caustic soda before dyeing is suggested as a means of minimizing streaks and resist spots. Fiber G (viscose) also has greatly reduced dye affinity, but is dyed reasonably well by a few selected developed and vat colors; most dark shades crock badly. With regard to ordinary viscose it is stated that it would seem unnecessary to dye

fabrics with colors of greater resistance to washing than the fabric itself. Greater dimensional stability in washing is needed with viscose fabrics. Two methods, still in the experimental stage, of applying vat dyes to rayon fabrics by padding methods are described.

Sulfur Dyeing

Some tendering and non-tendering influences commonly present in sulfur dyeing. Southeastern Section AATCC. *Am. Dyestuff Reptr.* **33**, P521–3 (Dec. 4, 1944).

Experiments have been carried out with sulfur black and blue dyes which indicate the following: (1) all standard sulfur blacks on the market exhibit practically the same tendering tendency toward cotton on which they are dyed under similar conditions; (2) the age of the dyes (at least up to 15 years from time of manufacture) does not affect tendering action; (3) the presence of free sulfur in the dyeing does not accelerate damage to the cotton; (4) the presence of iron as an impurity in sulfur black dyes accelerates tendering; (5) iron as an impurity in commercial sodium sulfide will tender cotton somewhat in a concentrated bath without dye being present; (6) metals ordinarily in contact with the sulfur dye bath have no effect in tendering; (7) different types of dryers have varying influence on tendering; (8) some oxidizing baths, such as chrome and acetic acid, may result in later tendering unless the goods is properly finished; sodium perborate appears to be safe; (9) sulfur-dyed goods are very resistant to tendering as long as alkali remains in them.

Color Yield in Printing

Factors affecting color yield in vat color printing. Rhode Island Section, AATCC. *Am. Dyestuff Reptr.* **33**, P502–9 (Dec. 4, 1944).

Experiments designed to hold constant the known factors influencing the color yield in vat color printing and to determine the nature and mode of action of other variables affecting this yield are described in detail. The following characteristics of the thickener were found to affect materially the color value of finished print: (1) degree of dispersion of the vat pigment in the paste, (2) degree of penetration of the paste into the fabric, (3) extent of protective colloid action, if any, of the paste (preventing dye from attaching itself to the fiber), and (4) physical quality of paste determining amount actually delivered to the fabric. The paste must inhibit the reagglomeration of the color particles which markedly reduces color yield even though it does not decrease the dye content of the fiber. With different types of thickeners, no correlation was found between color strength and viscosity of the paste. A mixture of starch, British gum, and bentonite (clay) materially prevented reagglomeration, reduced penetration slightly, and had no protective colloid action, resulting in a substantially increased color yield.

Mechanism of the Mordanting of Animal Fibers

Mechanism of the mordanting of animal fibers. C. H. Giles. *J. Soc. Dyers and Colourists* **60**, 303–14 (Nov. 1944).

The literature published during the past seventy years dealing with the mordanting of animal fibers is surveyed. Subjects discussed include the historical development of mordanting processes, the outlines of the mechanism of mordanting, and the nature of the various compounds present on the fiber, both before and after dyeing. Chromium mordants are dealt with extensively, whereas the use of other metals is considered more briefly. Wool is usually mordanted with potassium or sodium dichromate, either alone, with sulphuric acid, or with an organic reducing agent—e.g., oxalic acid or tartaric acid. The fiber absorbs chromic acid, which in the cold remains combined as such with basic groups of the protein molecule. Under practical conditions, however, when mordanting takes place at a high temperature, the steady change in the color of the fiber to an olive or greenish shade is an indication of reduction of the chromic acid. This change is brought about by an attack on the sulfur of the cystine bonds in the fiber, leading to the formation of compounds in which chromium is in the cation. The dyed fiber probably contains chromium atoms linked with both dye and wool molecules. Mordants containing the metal in the cation—e.g., chrome alum, potash alum, or ferrous sulfate, which are now of little practical importance, are hydrolyzed by the initial absorption of free acid by the wool fibers, and the metal is then absorbed in the form of a cationic complex.

Linseed Oil on Cotton Duck

The effect of linseed oil on cotton duck and its relationship to the performance and durability of railway wagon sheets. M. S. J. Twiselton. *J. Soc. Chem. Ind.* **63**, 220–3 (July, 1944).

The effect of pigmented linseed oil coatings on cotton duck treated with various rot-proofing agents is studied. Details are given of an investigation into the behaviour of oil-dressed rot-proofed cotton ducks prepared under various conditions of drying. The results show that the Shirlan NA-p-nitrophenol rot-proofing agent, in conjunction with a bauxite residue-linseed oil dressing, is superior to other processes examined in having the minimum damaging effect on the cotton. What are considered to be the salient points in the mechanism of attack on cotton by linseed oil in the presence of rot-proofing agents are indicated and theoretical support is given.

Shrinkage Control

Shrinkage and felting control with Lanaset. J. E. Lynn. *Am. Dyestuff Reptr.* **33**, P554–9 (Dec. 18, 1944).

Woolen goods shrinking less than 1% warp and 1% filling after 15 to 20 launderings could correctly be termed "shrinkproofed." No known

treatment yet fulfills this goal—present methods affect only shrinkage control. Methods used abroad, especially chlorination, have not been particularly successful in the U.S.; moreover, their action depends upon wool degradation and rigid controls are necessary. Methylol or an alkylated methylol melamine resin monomer (trade name: Lanaset) has given outstanding results in controlling wool shrinkage, and is unique among resinous materials in this respect. The control obtained will last for at least 10 Federal Spec. CCC-T-191A launderings. It can be simply applied by impregnation in a pad mangle, drying and curing, followed by washing to remove excess resin and accelerator. The curing which is best carried out separately from the initial drying is usually run at 290–310° F. It has been shown that the heat necessary for curing will not damage the wool; even a short passage at 400° F caused no noticeable damage. The slight yellowing and slight harshening occasionally observed are readily corrected. Heating undyed wool for 140 minutes at 302° F did not change the dyeing rate appreciably. To get optimum results with Lanaset the oil content of the untreated fabric should be as low as possible, preferably below 0.5%, when properly applied melamine base resins do not detract from the desirable properties of the wool. Application of the method to forms of wool other than fabric is not yet entirely successful. A further effect of melamine resins on wool is said to be the elimination of bleeding of many dyes.

Shrinkproofing

The chlorination of woven union fabrics. C. S. Whewell and B. Canket. *J. Soc. Dyers and Colourists* **60**, 302–3 (Nov. 1944).

It is known that mixed fabric containing Fibro (viscose staple) or Rayolanda X ("basified" viscose staple) shrinks considerably on milling unless the percentage of nonfelting fibers is high. When more than 25% of wool is present a shrinkproofing treatment is necessary for certain types of fabric. Series of tests have been made to determine the effect of wet chlorination (as applied to all-wool fabrics) upon a variety of wool-Rayolanda and wool-Fibro union fabrics. Three series were run, using increasing amounts of chlorine. Results showed that the lightest chlorination though insufficient to produce satisfactory shrinkproofing was more effective on union than on all-wool fabrics; this is interesting in view of the fact that untreated union fabrics frequently shrink more than the corresponding all-wool fabric. The more severe chlorinations produce satisfactory shrinkproofing of the union cloths; Rayolanda is less susceptible to the treatment than Fibro is.

Steam Scouring

Elimination of kier or jig scouring by alkaline steaming. G. P. Vincent, A. L. Dubeau, *et al. Am. Dyestuff Reptr.* **33**, P536–8 (Dec. 4, 1944).

A continuous alkaline steaming process whereby kier and jig scouring can be eliminated regardless of subsequent treatment is described. Plant-scale runs have shown that by this method a good dye bottom can be obtained on herringbone twill or poplin in less than one hour. The construction and operation of the continuous steaming chamber is discussed. Its features include a steam-tight chamber 60 feet long through which a conveyer passes and upon which the cloth is stacked (about 1 foot high) in loose folds, in open width. Delicate fabrics may be handled without danger of distortion. The steamer may also be used for any of the hot bleaching processes. Plant trials have been successful using the hypochlorite-activated Textone bleaching method (at room temperature) following a single alkaline steaming. By using the steamer (at 120–160° F) for activation bleaching a high white may be obtained in 30 minutes; when acid-Textone bleaching is used the steamer temperature is 212° F and a 10-minute steaming suffices.

FIBERS: YARNS: FABRICS MECHANICAL PROCESSES

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Fiber Bonding

The treatment of cotton roving and yarns with bonding agents. H. M. Chase. *Am. Dyestuff Reptr.* **33**, P474–8 (Nov. 6, 1944).

The ideal yarn would have the following characteristics: (1) all fibers would be alike and of maximum strength per cross-sectional area; (2) each fiber would bear its proportional share of the load; (3) rupture would occur at the breaking strength of the fibers (no slip); and (4) the yarn would be of perfectly uniform cross section throughout. Chemical finishing shows great promise in overcoming some of the defects of natural fibers and in more nearly approaching the ideal yarn. It has been found that yarn strength may be increased 70 to 80% by stretching the yarn, while it is in a plastic condition, almost to the breaking point and then setting a plastic bonding material on the yarn at this stage; the residual elongation thus obtained may be in the order of 1 percent—i.e., too low for certain purposes. It was found possible to increase the strength of tire cord 50—60% and still have the specified stretch of 6–7% at 10 pounds by means of a dual process, first stretching and setting the cord, then rewetting and restretching, so controlling the tension in the second step that the cord is left with the desired elongation. (The second stretching causes only a 10–15% loss in the originally increased strength, which proves that the initial stretching and setting produce a fundamental change in the character of the yarn.) A further refinement of the fiber bonding technique has resulted in the production of high-strength industrial yarn from roving without the use of a spinning frame; by chemically treating, stretching, compacting, and curing the roving a yarn is produced which has much greater strength than an eight-ply yarn of comparable yarn number.

Synthetic Fibers

A survey of the synthetic fibers. Wm. D. Appel. *Am. Dyestuff Reptr.* **34**, P21–P24 (Jan. 15, 1945).

The available data on the synthetic fibers have been summarized and compared with similar data for cotton, wool, and silk. They have been assembled from the viewpoint of the use of the fibers as engineering materials. It is pointed out that in many instances comparable data are lacking and that there exists a real need for a systematic comparative study of all fibers in a single laboratory under well-defined conditions. Information concerning composition and structure, dimension and shape, and physical properties is presented in a series of tables; the physical properties discussed are tensile strength, extensibility, resilience, elasticity, stiffness, and density. Published data on chemical properties are limited and less comparable; the effect of heat and moisture on many of the fibers is described. The information assembled is conveniently arranged and should prove a convenient reference source.

This month's progress in synthetic fibers. Anon. *Silk and Rayon* **18**, 1209–11 (Nov. 1944).

Brief reviews are given of the following: metallized direct dyes, E.P. 561, 117; determination of hemicellulose in viscose rayon (Barton and Prutton, *Ind. Eng. Chem. Anal. Ed.* **16**, 429 (1944)); spinning viscose rayon at higher speeds, E.P. 554, 689; and the manufacture of new maleic acid interpolymers, E.P. 562, 092.

Synthetic fibers in military and postwar fabrics. H. W. Rose. *Am. Dyestuff Reptr.* **34**, P26–P29 (Jan. 15, 1945).

Synthetic fibers have come into prominence in this country on a technical and scientific basis, whereas their growth in the totalitarian states has been due to national policy. They have played an important part during the war in conservation, in economic warfare, and in the manufacture of military fabrics to meet technical specifications and special requirements. Some of the uses for which synthetics have proved particularly valuable are escape, cargo, mine, fragmentation, and flare parachutes, tire cord, self-sealing gasoline tanks, shoe-laces, sleeping and shelter equipment, special garments, mosquito head-nets and screens, insulation, and powder bags. The characteristics which fit the fibers for these uses are discussed. Glass fabric has been used in a unique fashion as a combination heat shield and reflector in flare chutes. Postwar possibilities for fibers developed as a result of war needs are suggested; for example, civilian items such as rugs, upholstery, and laminated plastics may be found for high tenacity viscose yarns; synthetic rubber-coated glass fabric may provide fireproof awnings. Use of synthetic fibers, such as sewing threads for a number of war uses, is being intensively investigated and promises to lead to an expanding use of synthetic fibers as threads in peacetime.

Synthetics in Knitted Fabrics

The use of synthetic fibers in knitted fabrics. C. W. Bendigo. *Am. Dyestuff Reptr.* **34**, P34–P36 (Jan. 15, 1945).

It is reported that multifilament Velon yarn is being developed for hosiery. The specific gravity of Velon is 1.7 compared with about 1.5, 1.3, and 1.1 for viscose, acetate, and nylon respectively; 100-den. Velon has been knitted full fashioned as fine as 51 gage and is also said to be suitable for circular knit hosiery because of its thermoplastic properties. At present this yarn can be colored only in the form of the spinning mass before filament formation. Nylon yarn will probably be produced in the form of 15-den. monofilament and knitted on machines as fine as 66 gage. Nylon will also continue the trend toward circular knit hose shaped by heat. Developments in sizing and oiling synthetic yarns for knitting are discussed. It is suggested that there will be an increasing demand for thrown synthetics for warp knitting; this is one method of achieving dullness and richness in such fabric as jersey. The toe-to-top method of manufacturing staple is said to answer the need for a soft twisted knitting yarn of good strength and makes 100% spun synthetic knit goods practicable.

Textile Fibers

Textile fibers—new and old. J. Wilcock and H. Kennedy. *Fibers, Fabrics, and Cordage* **11**, 376, 378–9, 381 (1944) (through *Chem. Abstr.*, 1944, **39**, 624[1]).

Fibers from egg, milk and corn, metal and glass fibers, nylon and vinyon, Saran, Bubblfil and Celta, coating yarn with plastic, ropes and fabrics from glass, war as an inducement to synthetic-resin research, synthetic substitutes for natural fibers, "lineny" type rayon, and a place for natural fibers are discussed.

The story of modern textile fibers—viscose rayon. Anon. *Silk and Rayon* **18**, 1101, 1102, 1104, 1107 (Oct. 1944).

A review of viscose rayon technology. Curves are given showing (1) the degradation of cellulose during conversion to alkali cellulose and to viscose spinning solution as measured by its viscosity in cuprammonium solution, (2) the effect of time of steeping in alkali and (3) of temperature in formation of alkali-cellulose, (4) the influence of temperature and (5) of length of time on the ageing of alkali-cellulose.

Knitting

Cotton knitting yarn. Factors influencing its knittability. South Central Section AATCC. *Am. Dyestuff Reptr.* **33**, P524–8 (Dec. 4, 1944).

The characteristics necessary in cotton yarn for optimum knitting are discussed. Any process destroying the natural lubricity of the yarn

makes for inferior knitting; mercerizing is an exception, however, because of the smoothness and better fiber alignment acquired in this treatment. The twist in knitting yarn should be soft enough so that the fibers are in a more or less relaxed position; this also assists in chemical processing. Combed yarns are superior to carded yarns because the greater parallelism adds softness. Dyeing, when carried out with proper auxiliary agents for wetting out and for lubricating in the final baths, does not produce poor knitting yarns. Stripping chemicals destroy softness and lubricative qualities to a large degree and the yarn must be treated to compensate for this action. Peroxide bleaching, depending upon the pretreatment and the final washes, can produce yarn ranging from satisfactorily soft to very harsh. The terms "fiber-fiber lubricity" and "yarn-metal lubricity" are suggested as useful in discussing the factors contributing to knitting quality; the former characteristic is a necessary quality on the ribber and the latter on the knitter. A series of tests is described in which yarns of various characters are treated with a number of common lubricants (both fiber-fiber and yarn-metal types) and are subjected to standard knitting. It is shown that softness alone is not a criterion for a suitable knitting yarn but simply a necessary factor.

Knitting Shrinkage

The influence of knitting structure on the shrinkage and finishing of hosiery fabrics. W. A. Dutton. *J. Soc. Dyers and Colourists* **60**, 293–301 (Nov. 1944).

Due to military requirements machines of many types and gauges have been employed to make garments of the same standard size and weight. In order that all of these garments may meet the same standards of unshrinkability a considerable variation in knitting structure (i.e., courses per inch, and tightness or slackness of loops) is necessary; the result of variation in knitting structure on the dimensional stability in subsequent processing—e.g., storage, soaking, scouring, chlorination, and boarding—of both untreated and shrinkproofed fabrics has been investigated. It is shown that with fabrics not treated the shrinkage due to felting varies according to the stiffness of the knitting; even when chlorinated yarns are used the effect of slack knitting is to give considerable shrinkage. Lightly chlorinated yarn slack knit will start to felt rapidly once the surface fibers are removed by washing; on the other hand, when chlorinated fabric is slack knit the penetration is better and the felting is less than it would be if the fabric were tightly knitted; however, slack knit fabric may lose shape badly (even if the total area is not decreased) when it is washed, because of lack of cohesion between loops. It is pointed out that knitting and finishing are interdependent and that the knitter and finisher must cooperate closely to obtain the best results.

Processing Hosiery Yarns

Effect on the appearance of hosiery made from viscose, Bemberg, and acetate yarns subjected to variations in tension and steaming during throwing. Philadelphia Section, AATCC. *Am. Dyestuff Reptr.* **33**, P518–21 (Dec. 4, 1944).

Because of the sensitivity of rayon it is easy to produce undesirable effects in certain critical stages in in the processing of hoisery yarns. Improper handling with respect to tension can cause shortened loops in the finished product with consequent variation in the reflecting surface (streaky effects) and general irregularity of stitch formation. Stress-strain diagrams on viscose, Bemberg, and acetate hosiery yarns are included, showing the effect of stretching, and stretching followed by relaxing in water and steam. It is noted that all of the yarns which had been stretched only retained a permanent set, the amount of which depends on the degree of stretch and the amount of moisture present at the time of stretching. Tensions sufficient to produce a permanent set are not uncommon in reeling, winding, etc., being due, for example, to dirty or worn guides, non-uniform conditioning, non-free-running spools, and the like. Both water and steam appear to relax viscose yarn completely regardless of how much it has been stretched. Bemberg and acetate are completely relaxed only when the previous stretching is relatively low; conversely, this accounts for the fact that viscose is more susceptible to injury in the relaxed condition. Steaming, used by the throwster for setting twist, is also useful for removing irregularities introduced previous to that operation; however, if not carried out uniformly it will itself cause variation in dye absorption.

MISCELLANEOUS

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Color Vision

Standard response functions for protanopic and deuteranopic vision. Deane B. Judd. *J. Research Natl. Bur. Standards* **33**, 407–37 (Dec. 1944).

The color matches set up by the normal observer can be predicted satisfactorily by three functions of a wave length defining the ICI standard observer. It has been found possible by a transformation of coordinate system to express these three functions in such a form that two of the three pairs also represent the color matches of the two recognized types of red-green-blind observer, the protanope and the deuteranope, within the rather small uncertainties to which they are known. The remaining pair of functions represents, within the comparatively large uncertainties to which they are known, the color matches of the tritanope, a more rare type of observer who confuses reddish blue with greenish yellow. These three functions, therefore, serve to relate the color matches made by dichromats to those made by normal trichromats, and so

make conveniently accessible the color confusions of average dichromatic observers. The use of these three functions in the solution of problems arising in the design of tests for color-blindness is illustrated by the solution of three such problems, and their connection with theories of color vision is discussed.

Cellulose-Bearing Materials of India

Technical survey of cellulose-bearing materials of India. Nazir Ahmed and M. G. Karnik. *J. Sci. Ind. Research* **2**, 275–90 (1944) (through *Chem. Abstr.*, 1944, **39**, 184[7]).

The chief object of this investigation was to determine the α-cellulose content of cellulose-bearing materials of India under the optimum conditions of kier boiling and bleaching. Four processes were used for the wood samples: the soda, sulfate and sulfite and Na_2SO_3 alone; of these Na_2SO_3 gave the best results; addition of Na aluminate increased the α-cellulose content but also increased the ash content. The various stalks were pulped under high pressure in an autoclave. Cooks are reported for bamboo (*Dendrocalamus strictus*), thorny bamboo (*Bambusa arundinacea*), kamti and eetta bamboos, reeds, salai wood (*Boswellia serrata*), karai wood (*Sterculia urens*), fir, spruce, cotton stalks, maize stalks, castor stalks, sunn hemp stalks, linseed, rice and wheat straws, kazi grass, coconut husks, bagasse, rametha bark, aloe leaves, jute, hemp, and cotton. Salai and karai woods did not give satisfactory results (short fibers and low α-cellulose content). Cotton, castor, and sunn hemp stalks stand next to bamboo in the order of merit. Wheat straw was better than linseed and rice straws and has about the same α-cellulose content). Cotton, castor, and sunn hemp stalks stand next to bamboo in the order of merit. Wheat straw was better than linseed and rice straws and has about the same α-cellulose content as bagasse and kazi grass. The yield from rametha bark was low and the ash content is high. It is emphasized that these experiments were carried out with the simplest possible equipment and only give approximate values.

Drying with Radiant Heat

Infra-red drying. Some experiments and results with gas-fired equipment on high-speed baking of paint finishes and other drying processes using radiant heat. F. L. Atkin. *Gas J.* **244**, 371–4, 402–4, 407–9, 413, 415 (1944); *Gas World* **121**, No. 3142 Ind. Gas. Suppl. 101–8; No. 3146 Ind. Gas Suppl. 118–23 (through *Chem. Abstr.*, 1945, **39**, 229[7]).

The article includes descriptions of experimental work on the factors influencing rapid drying by radiant heat, of tests on the drying of various articles and other heating processes, and of radiant tunnel installations. The following conditional premises for successful infrared drying are suggested: (1) a regular and controllable movement of the articles through the tunnel by mechanical means; (2) with a given rate of progression, similar thickness for the articles; (3) the same type and color of paint for the articles stoved at the same time and (4) preferably use of a paint with rapid drying properties. Problems in the design of infrared equipment and for manufacturers of paints for infrared drying are outlined.

Insect Damage

Prevention of damage to paper, books and documents, by insect pests. F. D. Armitage. *Patra J.* **8**, 40–9 (Nov. 1944) (through *Bull. Inst. of Paper Chem.*, **15**, 164, Jan. 1945).

Because books and paper are often stored under very adverse conditions in Great Britain at the present time, the author discusses five representative types of insects which are liable to attack cellulosic materials: the silver fish, the cockroach, the so-called book louse, the drug store beetle, and the common clothes moth. He describes the life history of each of these pests, the way in which they become harmful, and methods of prevention. Old warehouses should be carefully inspected for worm-eaten timbers and dirty floors before stocks of paper or valuable books are stored in them; if in a bad state, they should be washed thoroughly and disinfected (fumigated) by an expert prior to their use.

Printed Fabrics in Laminates

A new field of melamine. Anon. *Modern Plastics* **22**, 105–7, 182, 4 (Jan. 1945).

Exact reproductions of colored designs can be achieved in laminates by impregnation of printed paper or fabric surface sheets with melamine resins. This is done in a hat-type press using a pressure of not more than 250 p.s.i. Exact details as to the amount of resin, time, and temperature cycles are not given. Dyes that are sunfast and tub-fast and closely woven fabrics produce much superior designs as to fineness of detail in the finished laminate. Inks other than those with linseed oil base give best results for paper drawings or designs.

Moisture in Textiles

The moisture relations of textiles. A survey of the literature. P. W. Carlene. *J. Soc. Dyers and Colourists* **60**, 232–7 (Sept. 1944).

A comprehensive survey is made on the work done on the sorption of water by textiles; the most important of this work has occurred in the last twenty years. Special attention is given to the classic researches on the moisture absorption of cotton carried out by Urquhart and Williams, and to the absorption-desorption hysteresis effect which is noted in all textile fibers (with the possible exception of nylon and other newer synthetics). Urquhart's explanation of this hy-

steresis is discussed. Further published work concerned with cotton cellulose by such investigators as Pierce, Walker, Tankard, Babbitt and Assaf is summarized. The moisture relations existing in the case of rayons, wool, silk, jute, and nylon are also reviewed. Included in the survey are: the influence of moisture on the elastic properties, the swelling, and the rigidity of the various fibers, the effect of processing, i.e., drying, mercerizing, bleaching, dyeing, and possible concurrent chemical attack, on the absorbency of textiles.

Moisture Absorption

The moisture relations of textiles. A survey of the literature. P. W. Carlene. *J. Soc. Dyers Colourists* **60**, 232–7 (Sept. 1944) (through *Bull. Inst. Paper Chem.*, **15**, 146, Dec. 1944).

Most of the work of outstanding importance on the moisture absorption of textiles has been published during the last twenty years. The author reviews the results of the more important investigations during this period under the headings: the absorption and desorption of water by fibers; the influence of moisture on the physical characteristics of textile fibers; and the effect of processing upon the moisture absorbency of textiles. 56 references.

Modification of Silk

Chemical methods for modifying the properties of silk. 3. Methylation treatments. Anon. *Silk and Rayon* **18**, 1236–7 (Nov. 1944).

A review of the work on modification of real silk by methylation. The fibroin may be acetylated by means of ketene or acetic anhydride or may be methylated by treatment with diazo-methane, or with dimethyl sulfate under alkaline conditions. It cannot be said that the treated silk is improved in any material respect.

Mothproofing Furs

The mothproofing of furs. D. Traill, A. McLean, and H. H. S. Bovingdon. *Chemical Ind.* **32**, 284–6 (Aug. 5, 1944).

Two types of materials are available for permanently mothproofing keratinous substances. These are inorganic salts including hydrofluoric acid derivatives and organic compounds similar to acid-milling dyestuffs. Details of experiments are given to test the efficacy of mothproofing protein fibers by treatment with a saturated solution of sodium chloride which contains small amounts of formaldehyde and hydrochloric acid. This treatment has a marked deterrent action on oviposition and larvae development. The process lends itself better to fur treatment than to wool in general.

Plastics for Coatings

A new plastic with textile possibilities. J. Wakelin. *Silk and Rayon* **18**, 1116 (Oct. 1944).

Ethylene polymers which soften between 100° C and 115° C may be used as a coating for paper or fabrics. It is applied by impregnation from solution or calendering. This gives a water- and chemical-proof coating which is inert and odorless. Instructions are given for treating window-shade material and transparent cellulose foil with polymerized ethylene. Chlorinated polythenes (known as "halothenes") are non-inflammable as well as waterproof and flexible and have been patented for doping aeroplane-wing fabrics.

Plastics and Textiles

Plastics and their future in the textile industry. H. Jones. *J. Soc. Dyers and Colourists* **60**, 225–32 (Sept. 1944).

Techniques such as co-polymerization, plasticization, and the use of catalysts, accelerators and lubricants, which may be used to control the properties of plastic materials to increase their applicability in the textile field, are discussed. Polymerization may be effected either in the form of the pure substance or in the form of an aqueous emulsion; in the latter case the use of an inflammable solvent is avoided, and much greater resin concentration is obtained for the same viscosity than when in solution (formaldehyde type resins in initial stages of condensation are soluble in aqueous media). In many cases the chemical reactivities of resins may be utilized to broaden their usefulness; e.g., vinyl acetate polymers may be hydrolyzed to give polyvinyl alcohols used as water-soluble sizings and coatings. The modern linear polymer rayons are discussed with predictions as to their possible development. Cellulose triacetate and the higher esters of cellulose, cellulose ethers, benzyl cellulose and the resin-modified cellulose type rayons, polyamides and polyesters, are said to have particular promise. It appears that the German fiber PeCe (which contains 60% chlorine) is made from 100% polyvinyl chloride and is chlorinated subsequent to polymerization; its advantage over Vinyon is acetone solubility and a higher softening point. The use of plastics as bonding agents for fibers and fabrics, for binding pigments, for textile finishes and for coated fabrics is reviewed. Thermoplastic resins, on account of the large molecular size of the polymers, are of little use in anti-crease processes, and their polymerization *in situ* is impractical because of the low boiling point of their monomers; however, new and less volatile materials may be anticipated, e.g., vinyl naphthalene, or methods of polymerizing without heat treatment.

Reclaiming Nylon

A new chemical method for reclaiming nylon. Anon. *Chem. Ind.* **55**, 376–8 (Sept. 1944).

A process is described for reclaiming nylon chemicals from a mixture of discarded nylon hose, nylon fiber

waste and clippings, as well as drippings from spinnerets and other machines. This waste is put in a sulfuric acid solution in a hydrolyzer and boiled. On cooling adipic acid crystallizes out and is separated by centrifuging. This process is repeated three times. Further purification follows by use of charcoal, centrifuging, and recrystallization. The diamine is recovered by treatment with lime, then centrifuging, and distilling. The reclaimed diamine and adipic acid are combined in the correct proportion, clarified with charcoal, and nylon again synthesized.

Organization of Research Department

An industrial research department. D. E. Chambers. TEXTILE RESEARCH **14**, 359–65 (Nov. 1944).

The essential factors to be considered in the organization and operation of an industrial research department are discussed. As a basis for the description the research laboratory of the General Electric Company was used.

Polyethylene

Polyethylene. Howard H. Irwin. *India Rubber World* **111**, 65–6 (1944) (through *Chem. Abstr.*, 1945, **39**, 439[7]).

A review of the history, preparation, chemical and physical properties and uses of polyethylenes. Sixteen references.

Thermostats

The practical side of fine temperature control. R. Barrington Brock *and* L. T. Townson. *Chem. and Ind.* **41**, 358–60 (Oct. 14, 1944).

The problems of fine temperature control fall under four heads: stirring, heating, temperature control and heat losses. "Straight line stirring" is described and is recommended because errors due to eddies and vortices are avoided. Heater lamps are considered the best mode of heating where practical, but metal-incased heaters are satisfactory for many purposes. The authors maintain that it is incorrect to state that either the bimetallic or the toluene thermoregulator is the better. It is suggested, however, that the bimetallic is probably better for air baths and the toluene bulb for liquid baths. Thyratron tubes are superior to ordinary relays for systems in which there is very frequent making and breaking of the circuit. High heat losses cannot be properly compensated and must be kept to a minimum. However, the use of glycol for this purpose in high temperature baths is condemned. This paper is a summary of a lecture and omits many important details.

Properties of Synthetic Rubbers

Comparison of natural and synthetic rubbers. Anon. *Chem. Ind.* **55**, 738 (Nov. 1944).

Natural rubber, neoprene, thiokol, buna-N, buna-S and butyl rubber are compared qualitatively as regards 34 physical and chemical properties. Most of the properties tabulated may be classed as being of the "resistance to" type. The materials are graded as being excellent, good, fair or poor.

Ultraviolet Light

Seasonal variations of ultraviolet energy in daylight. Matthew Lurkiesh, A. H. Taylor, and G. P. Kerr. *J. Frank. Inst.* **238**, 1–7 (July, 1944).

The intensity of solar energy in the ultraviolet region is dependent on the absorption band of ozone which filters out wavelengths shorter than 2950Å. During 8 months of the year more than half of the erythemal ultraviolet energy is contributed by a clear sky. During mid-summer there are only a few hours during the middle of the say when the value from the direct sun is greater than that from a clear sky. The total erythemal ultraviolet energy received during the six months from October through March is only 10% greater than that received during the month of June or July. The amount received during December is only 1% of that received for the entire year. Data based on observations at Cleveland, Ohio. Maps are included showing the percentage of possible sunlight received by various localities of the U. S. in winter and summer.

Wet Strength Paper

A new market for new chemicals: wet strength paper. K. W. Britt. *Chem. Ind.* **55**, 734–7 (Nov., 1944).

Trends in the paper industry presage an increasing use of wet-strengthening chemicals in the manufacture of wrapping and packaging materials, absorbent and cleansing papers, and in the use of paper products for structural materials. Chief of the resins used today are urea-formaldehyde and melamine-formaldehyde which are deposited upon the fiber from aqueous solution and polymerized *in situ*. This polymerization is accomplished by heating in the presence of a catalyst or by allowing the paper to cure during a storing period. The resins used are three types: those suitable for impregnation of paper sheet, such as dimethylolurea; those for stock treatment before sheet formation, and those that may be used either way. The degree of polymerization of the resin largely determines whether it may be used in the beater or applied to the finished sheet. The last page of this article is printed on wet-strength paper.

Chromatographic Adsorption

Chromatographic adsorption. Ernest L. Eliel, Ed. *J. Chem. Ed.* **21**, 583–8 (Dec. 1944).

A résumé of chromatographic adsorption, with special emphasis on apparatus and technique used. A brief history, a theoretical discussion, and important applications in organic and inorganic chemical work are given.